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Dow Chemical Building 703 Incinerator Exhaust and Ambient Air Study

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16. ABSTRACT

The purposes of ~~this~~ the study were: (1) determine the levels of dioxins and other toxic compounds in ambient air near the Dow Chemical Midland plant; and (2) determine the levels of dioxin and other chemicals in the Building 703 incinerator exhaust gas, wastewater, and ash, under normal operating conditions. The ambient air study included positive findings of low levels of dioxins at air monitoring sites near the plant fence line and at the site located in the city, ranging up to 0.0004 ug/m^3 for the less toxic forms. 2378-TCDD, the most toxic form of dioxin, was found on one day at a site near the Dow Chemical fence line at a level of 0.000005 ug/m^3. The proportions of various dioxin compounds found in this study were similar to those found in studies of air in incinerator areas and soils sampled by USEPA in 1984. The incinerator exhaust study found that various forms of dioxin were present on all three sampling days in the incinerator exhaust gases at concentrations between 0.005 and 0.125 ug/m^3. The study concluded there were no readily observable relationships between the incinerator temperature, pressure, air pollution control device and flow rates, and the levels of certain dioxins found in the exhaust during the three days of testing. However, there may be a relationship between the levels of dioxin fed into the incinerator and the levels of dioxin discharged.

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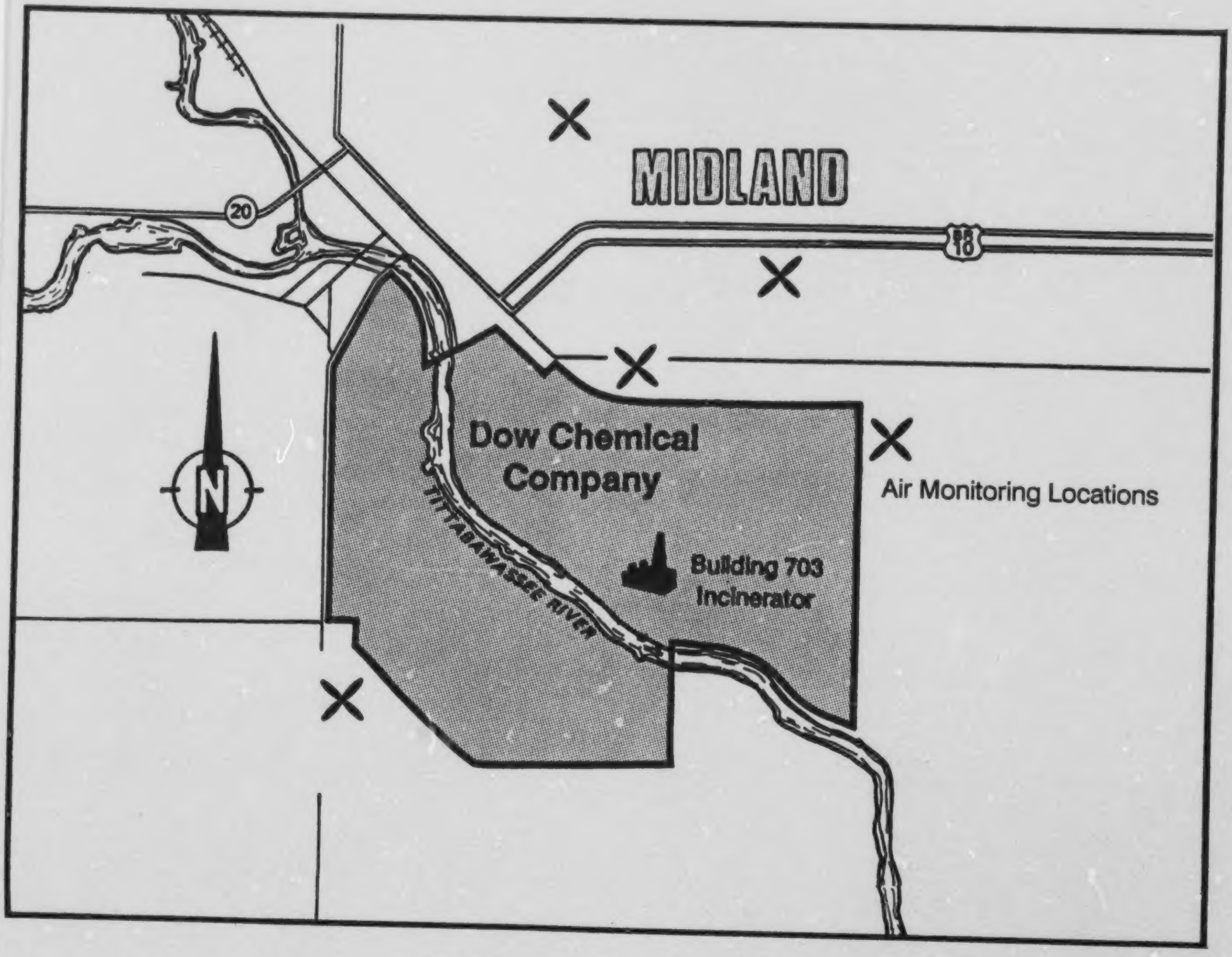
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DOW CHEMICAL BUILDING 703 INCINERATOR EXHAUST AND AMBIENT AIR STUDY

MARCH 1987

MARTIN G. TREMBLY
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U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION V
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Appendix C Analytical Procedures for PCDD/PCDF - Brehm Laboratory - Wright State University

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Appendix E Detailed Descriptions of Ambient Air Monitoring Equipment and Sampling Methods - Michigan Dioxin Studies - Midland, Michigan, Ambient Air Sampling Study

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Appendix J Detailed Discussion of Air Dispersion Modeling to Determine Point of Maximum Ground-Level Impact

I. INTRODUCTION

The U.S. Environmental Protection Agency (USEPA) Region V has conducted a series of comprehensive multi-media studies of dioxins and other toxic pollutants at the Dow Chemical Company Midland Plant, in the Tittabawassee River, and in and near the city of Midland, Michigan. The purposes of these studies were to determine current emissions and ambient levels of toxic pollutants and whether those levels warrant remedial action to minimize or eliminate public exposure and environmental risks. Soil sampling was conducted in Midland, at Dow Chemical, and at comparison and background sites during 1983 and 1984. Results from the soil study were released in April 1985.[1] Screening studies of surface water supplies, potable ground water, and brine operations at the Dow facility was completed in 1984 and 1985; the results of these studies were released in December 1985.[2] During 1984, samples were collected from Dow Chemical's waste incinerator; ambient air; Dow Chemical's industrial wastewaters; and Tittabawassee River sediments. In July 1986, Region V also released the results of comprehensive testing of Dow Chemical in-plant and effluent wastewaters, sludges, and Tittabawassee River sediments and native fish.[2a] This report presents the results of the hazardous waste incinerator emissions testing and ambient air monitoring.

On December 1, 1983, EPA published a Dioxin Strategy,[3] which provides a framework under which the Agency is to

- study the nature and extent of contamination of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2378-TCDD) and the associated risks to humans and the environment;

- implement or compel necessary cleanup actions at contaminated sites; and

- further evaluate regulatory alternatives to prevent future contamination, as well as disposal alternatives to alleviate current problems.

The Dioxin Strategy focuses on seven tiers of sources, ordered by decreasing potential for 2378-TCDD contamination. Combustion sources, including the Dow Chemical waste incinerator, comprise Tier 4. EPA published sampling and analysis plans in February 1985.[4] Facilities studied as part of the Tier 4 strategy were sampled at specific fuel and air input points; air, solid, and water effluent points; and selected surrounding sites for soils. This sampling and analysis model is reproduced as Table I-1. The Dow Chemical facility was studied consistent with the Tier 4 model, but on a separate schedule and with different field sampling and analytical teams.

In March 1983, the Michigan Department of Natural Resources (MDNR) requested that EPA conduct studies of the presence of dioxins and other compounds in various media in the Midland area. Responding to this request, the range of compounds selected for study in the incinerator emissions and ambient air studies was expanded from that shown in Table I-1 to include several which have estimated risks associated with respiratory exposure (see Tables V-1 and V-2).

TABLE I-1

TIER 4 DIOXIN SAMPLING AND ANALYSIS STRATEGY - JUNE 1984 DRAFT

Sample	Method	Recommended (Samples/Day)	Recommended Analyses	Total Samples[a] for Analysis
Inputs				
Precombustion Air	XAD-2	0-1	2,3,7,8-TCDD, Homologs, PCBs, TOCl Cl-phenols, Cl-Benzenes	1
Quench Water	Grab	0		0
Feed/Fuel	Grab (every 4 hours)	Daily Composite	PCDD scan, Cl-phenols, Cl-benzenes PCBs, TOCl	3[b]
Outputs				
Stack (before control)	MM5T	2 trains -Gaseous daily composite -Condenser rinse -Adsorbent resin -Particulate daily composite -cyclone catch -filter -probe rinse	2,3,7,8-TCDD, Homologs	6
Stack (after control)	MM5T	2 trains -Gaseous daily composite -Condenser rinse -Adsorbent resin -Particulate daily composite -filter catch -probe rinse	2,3,7,8-TCDD, Homologs	6
Bottom Ash	Grab (every 4 hours)	Daily Composite	2,3,7,8-TCDD, Homologs	3-6[c]
Ash from Control Device	Grab (every 4 hours)	Daily Composite	2,3,7,8-TCDD, Homologs	
Quench Water Effluent	Grab (every 4 hours)	Daily Composite	2,3,7,8-TCDD	3
Environmental				
Ambient Air	XAD-2	0		0
Surface Water	Grab	0		0
Soil	Boring	1	2,3,7,8-TCDD	1
			TOTAL	23-26

[a] Based on 3 sampling days.
[b] Analysis by contractor.
[c] May be combined daily composite.

II. OBJECTIVES

A. Incinerator Exhaust Study

The purposes of the incinerator emissions study were to

- determine concentrations and mass loadings of polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), chlorobenzenes, chlorophenols, and other chemicals in the exhaust gas, wastewater, and solid matter (ash and waterborne suspended solids) discharges from the incinerator, under normal operating conditions; and
- relate the compounds found in the discharged streams to the characteristics of liquid and solid wastes incinerated at those times.

B. Ambient Air Study

A companion study of the ambient air around the Dow Chemical facility was designed to determine the presence of PCDDs, PCDFs, and other semi-volatile and volatile compounds; to relate these findings to air emissions from the Building 703 incinerator; and to detect other compounds that may be emitted from other point sources or fugitive sources at the Midland Plant. It was originally intended that ambient air samples would be obtained on the same days as incinerator exhaust sampling. However, resource requirements of this work dictated that the monitoring programs be conducted separately.

III. SCOPE OF WORK

A. Incinerator Exhaust Study

Numerous process vents other than the Building 703 incinerator exist at the Dow Chemical Company Midland Plant. However, based upon the results of Dow Chemical self-monitoring of principal process vents in organic chemical production areas, the incinerator was determined to be the dominant potential source of PCDD and PCDF emissions in the plant. Therefore, sampling was limited to the incinerator.

Sampling occurred on August 28, August 30, and September 5, 1984. The conduct of this work is described in Section V and Appendix A of this report. A total of 267 samples were collected and analyzed in this portion of the study.

B. Ambient Air Study

For the purposes of the ambient air study, the range of compounds selected for sampling was expanded beyond PCDDs and PCDFs, to include other compounds which have demonstrated risks through respiratory exposure; compounds with chemical structures similar to these; and other compounds which may have been traceable to emissions from the Dow Chemical Company incinerator. This selection was based upon an evaluation of information available in files of the MDNR and USEPA, concerning plant processes, products, intermediates, and wastes generated. The target compounds included the following:

acrylonitrile
benzene
biphenyl
chlorobenzenes
carbon tetrachloride
chlorinated biphenyls
chlorinated phenols
chloroform
ethylene dibromide
ethylene dichloride
ethylene oxide
formaldehyde
methyl chloroform
methylene chloride
perchloroethylene
phenylphenols
vinylidene chloride

Constituents such as metals (arsenic, beryllium, etc.) were not included as the above-referenced file information indicated they were not involved in plant processes, while others, such as vinyl chloride, were not among the target compounds as no available sampling methods were applicable to them. However, compounds not appearing in the above list were reported when they were detected, subject to quality assurance limitations, as shown in the discussion to follow.

Ambient air was sampled at four locations near the Dow Chemical Midland Plant on 18 days between September 7 and 27, 1984. The conduct of this study and a discussion of results appears as Section VI of this report; a total of 353 samples were gathered and analyzed.

IV. FINDINGS AND CONCLUSIONS

A. Incinerator Exhaust Study

1. Tetra- through octa-CDDs and tetra- through octa-CDFs were detected in Building 703 incinerator exhaust on two of the three sampling days. On the third day, only TCDDs, OCDD, TCDFs, and PeCDFs were found in the incinerator exhaust. TCDDs were detected in the 5 to 45 ng/m^3 range, while TCDFs were found at 80 to 125 ng/m^3. Other PCDDs and PCDFs were found at levels typically lower than 10 ng/m^3. 2378-TCDD was not detected in Building 703 incinerator exhaust at detection levels of approximately 1 ng/m^3. A single finding of 2378-TCDD in combustion air drawn into the incinerator is reported.

2. The Modified Method 5 train chosen as the sampling apparatus for PCDDs and PCDFs and semi-volatile compounds trapped these compounds, frequently at or near the low detection limits desired in this study. However, the collection and retention efficiency of the Modified Method 5 sampler for PCDDs, PCDFs, and other semi-volatile compounds has not been validated. Therefore, the analytical results stated for PCDDs, PCDFs, and semi-volatile compounds should be considered minimum values. Analytical accuracy was acceptable for 79% of the samples obtained for the measurement of tetra- and penta-CDDs, which are of greatest concern with respect to possible health effects.

3. Comparisons were made of mass inputs of PCDDs and PCDFs in incinerator feed streams, scrubber and quench waters, and combustion air, with mass outputs in stack emissions, scrubber and quench waters, and collected ash. A clear relation between the mass of PCDDs and PCDFs in input streams and discharges was not discernible; however, higher concentrations of PCDDs and PCDFs in liquid wastes consumed in the incinerator appeared to translate into higher levels of PCDDs and PCDFs in incinerator discharge streams. The data suggest limited destruction in the incinerator of TCDDs and HpCDDs, somewhat higher destruction of PeCDDs, and mass transfer of HxCDDs and OCDDs from input streams to output streams. For PCDFs, the data suggest destruction of nearly 90% of the input TCDFs, and formation of other PCDFs, primarily HxCDFs and OCDF.

4. Analyses for suspected PCDD and PCDF precursors in the influent streams, such as PCBs and chlorinated benzenes and phenols, were not conclusive in establishing a relationship between these compounds and emitted PCDDs and PCDFs. However, the concentrations of certain semi-volatile compounds such as chlorobenzenes and chlorophenols in the incinerator exhaust, did appear to relate directly with emissions of PCDDs and PCDFs.

5. While extensive data on incinerator operating temperatures, pressures, air pollution control device water, and flow rates were obtained, there was no discernible relationship between any of these characteristics and exhausted PCDD and PCDF concentrations within the ranges encountered in this study.

6. Quality assurance results for semi-volatile and volatile compounds indicated analytical accuracy and precision problems, such that data for these compounds were of limited quantitative use.

7. On two of three sampling days, emissions of particulate matter from the incinerator exhaust were within the standard of 0.08 grain/dscf established for incinerators burning hazardous waste (40 CFR Part 264.343(c)). Values ranging from 0.0615 to 0.0842 grain/dscf were measured with a mean value of 0.0747 grain/dscf.

8. Wastes from a nearby Dow Corning Corporation facility were incinerated on all three sampling days; these wastes contained PCDDs and PCDFs (primarily the 1368 and 1379 TCDD isomers). Ash discharged from the Building 703 incinerator included silane and siloxane compounds most likely attributable to Dow Corning.

B. Ambient Air Study

1. No 2378-TCDD was detected on a first analysis of ambient air samples by a contract laboratory. However, reanalysis of two of the 15 sets of sampling media by the EPA Environmental Monitoring and Support Laboratory, Research Triangle Park, North Carolina, resulted in detection of 2378-TCDD at an ambient site near the Dow Chemical Company plant fenceline at a concentration of about 5 pg/m^3. Ambient air monitoring by Dow Chemical in 1983 and 1984 showed positive results for 2378-TCDD up to 0.2 pg/m^3. The Dow Chemical data were obtained with a glass fiber filter in the sampler.

2. Other homologues of PCDD and PCDF were detected with generally acceptable accuracy in the range of 0.1 to approximately 400 pg/m^3. These homologues were present in proportions similar to those found in previous studies of ambient air near incineration processes, and in soils sampled by EPA in the Midland, Michigan, area in 1984.

3. The modified high-volume sampler used for PCDD and PCDF sampling trapped the full range of PCDD and PCDF homologues. However, the collection and retention efficiency of the Modified Method 5 sampler for PCDDs and PCDFs has not been validated. Therefore, the analytical results stated for PCDDs and PCDFs should be considered minimum values.

It was found that lower-chlorinated homologues generally were trapped in the second-stage XAD-2 resin trap incorporated into the samplers, while higher-chlorinated homologues remained in or on the first-stage glass fiber filter. While no particle size data were obtained at this time, these findings strongly suggest that both sampling media should be exposed in series to ambient air to sample for the full range of PCDD and PCDF homologues. Also, it is implied that lower-chlorinated homologues may either attach to finer, more respirable particulate matter, or may be air-stripped from larger particles caught in the glass fiber filter. No particle sizing data were gathered at this time to test this possibility.

4. A sampler similar to that employed for PCDDs and PCDFs was effective in sampling for semi-volatile compounds in ambient air. While analytical precision was remarkably good in many samples for semi-volatile compounds, accuracy problems were frequently encountered.

5. Sources within the Dow Chemical facility, other than the Building 703 incinerator exhaust stack, such as process vents or fugitive emissions sources, may be responsible for the levels and diversity of semi-volatile compounds detected in ambient air around the plant. Principal semi-volatile compounds found in this study were trichlorobenzene (three isomers); tetrachlorobenzene (two isomers); 2,4-dichlorophenol; and 2,4,6-trichlorophenol, in ranges from approximately 10 to 1000 ng/m^3.

6. Sampling for volatile compounds in ambient air did not yield valid data due to failure of a contract laboratory to prepare and analyze field samples within acceptable sample holding times. However, the results obtained have been reported and interpreted within the limits dictated by quality assurance results.

V. DOW CHEMICAL MIDLAND PLANT BUILDING 703 INCINERATOR EMISSIONS STUDY

A. Facility Description

The Dow Chemical Company operates a hazardous waste incinerator at the Midland Plant identified as Building 703. The incinerator is located in the southwest quadrant of the facility (see Figure VI-1). Liquid wastes, tars, and containerized and loose solid wastes are incinerated at this site. Company records indicate about 200 tons per day of solid and liquid combustible trash and waste are burned on a typical day. Built as a rotary kiln burner in 1957, the incinerator has been augmented in succeeding years by addition of an afterburner section and air pollution control equipment including a quench tower, venturi scrubber, demister, and a wet electrostatic precipitator. The level of complexity and expected efficiency of this air pollution control equipment is generally greater than typically found at other municipal or hazardous waste incinerators in the United States. A schematic drawing of the incinerator and associated air pollution control equipment as currently configured appears as Figure V-1.

1. Waste Feeds and Incinerator Operational Characteristics

Waste feeds to the incinerator are as follows:

a. Liquid Wastes

Liquid wastes are delivered to the incinerator from an adjacent tank farm, or via direct burn systems in which individual truck or trailer loads of wastes are consumed. Three wastes may be burned simultaneously in separate feed nozzles. Two of these nozzles, identified as "BA" and "BB", are located at the head end of the rotary kiln, and each feeds wastes longitudinally at an average rate of three to four gallons per minute, atomized with steam. Waste feed nozzle "C" is placed in the afterburner section. The nozzle is directed approximately at a right angle to exhaust gas flow to induce turbulence in the firing zone. These wastes are air-atomized, and typically flow at an average rate of five to six gallons per minute. Combustion may be supplemented with natural gas at all three nozzles.

b. Low-BTU Liquid Wastes

Collected rainwater or surface runoff from within the liquid waste tank farm and handling area, and other contaminated water such as carbon adsorption bed condensates and runoffs from reaction vessel cleaning, may be fed to the incinerator as needed to modulate temperature fluctuations. This water flow is not continuous. As allowed by the MDNR[24], surface runoff is directed to the plant wastewater treatment system if the total organic carbon content is less than 100 parts per million.

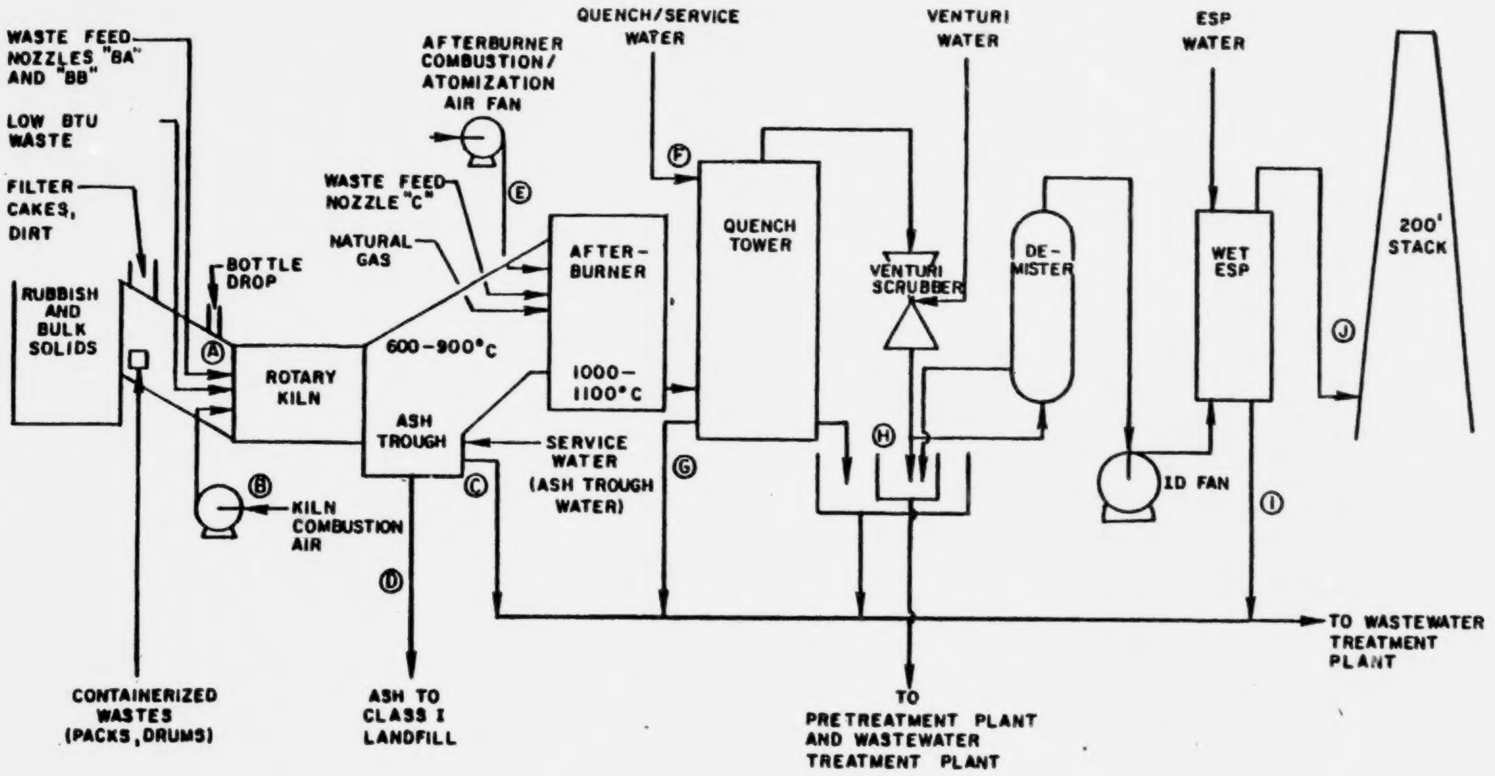

WASTE FEED NOZZLES "BA" AND "BB"
LOW BTU WASTE
FILTER CAKES, DIRT
RUBBISH AND BULK SOLIDS
BOTTLE DROP
Ⓐ
ROTARY KILN
Ⓑ
KILN COMBUSTION AIR
CONTAINERIZED WASTES (PACKS, DRUMS)
AFTERBURNER COMBUSTION/ ATOMIZATION AIR FAN
Ⓔ
WASTE FEED NOZZLE "C"
NATURAL GAS
600-900°C
ASH TROUGH
Ⓒ
Ⓓ
ASH TO CLASS I LANDFILL
SERVICE WATER (ASH TROUGH WATER)
AFTER-BURNER
1000-1100°C
QUENCH/SERVICE WATER
Ⓕ
QUENCH TOWER
Ⓖ
VENTURI WATER
VENTURI SCRUBBER
Ⓗ
TO PRETREATMENT PLANT AND WASTEWATER TREATMENT PLANT
DE-MISTER
ID FAN
ESP WATER
WET ESP
Ⓘ
Ⓙ
200' STACK
TO WASTEWATER TREATMENT PLANT

FIGURE V-1

SCHEMATIC DIAGRAM

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

c. Containerized Bulk Wastes

Various types of wastes in volumes too small or viscous to be handled in the liquid waste system, and solid wastes from laboratory operations, facility demolition and cleanup, are conveyed to the head end of the rotary kiln. One pack or barrel of containerized waste, weighing a maximum of 200 pounds, is introduced to the kiln every six minutes from a staging area in which several packs may be prepared sequentially for incineration. In a typical feed, a pack is advanced from a conveyor to the outside door of an air lock, the air lock door opens, the pack is pushed into the lock, and the outside door closes. Approximately 30 seconds later, the inside air lock door opens and the pack is dumped into the rotary kiln. At this time, another pack is moved to the outside air lock door, and the next pack in line is conveyed over a scale for weighing.

d. Miscellaneous Containerized Wastes

On occasion, small quantities, typically less than a few liters, of bottled liquid or tarry wastes may be introduced directly to the rotary kiln. A small air lock, or "bottle drop", is provided for this purpose, and company procedures provide that a person representing the area of the plant generating the waste be present during this operation.

e. Loose Rubbish

Loose solid waste generated in the Midland Plant is incinerated at Building 703. These wastes, consisting chiefly of paper, plastics, and wood, are dumped into a holding pit from whence a clamshell deposits batch quantities into a shredder. Shredded wastes are conveyed at a controlled rate of four to six tons per hour through an incline to the incinerator.

f. Other Wastes

Wastewater treatment plant solids were formerly fed to the incinerator along with loose rubbish. However, according to information provided by plant representatives, this waste stream was redirected to landfill disposal as of July 1984.

Combustion air is provided through two separate forced-draft fans in the rotary kiln and afterburner sections. Oxygen concentrations of at least 3 percent, but typically over 10 percent, are maintained in the afterburner exhaust. Combustion temperatures within the rotary kiln are designed to vary between 650° and 950°C, and between 1000° and 1100°C in the afterburner. The temperatures are maintained by cycling the combustion of wastes at the three nozzles and by selectively adding low-BTU liquid wastes to reduce high temperatures. Thus, temperatures within the rotary kiln may vary over a relatively large range within short time periods. However, temperatures within the afterburner fluctuate much more narrowly. Although rotary kiln and afterburner pressures are held slightly negative, usually between -0.1 and -0.3 inch of water, violent ignition of waste packs can cause momentary periods in which positive pressures are experienced.

Gas residence times within the incinerator have been calculated by Dow Chemical as one to three seconds in the rotary kiln, and approximately 1.5 seconds in the afterburner.[21,22]

2. Air Pollution Control Equipment

As indicated previously, the air pollution control system associated with the Building 703 incinerator consists of several components shown in Figure V-1. That figure also contains a schematic of water sources through each control device.

a. Quench Tower

Exhaust gases from the afterburner pass to the quench tower, in which gas temperature is reduced from about 1000°C to below 100°C. To accomplish this, a normal water application rate of 650 to 750 gpm is maintained. Secondary treated wastewater from the plant wastewater treatment system is the primary water supply. Water pumped from the company's Tittabawassee River intake may be used to supplement the primary supply. Water effluents from the quench tower split into two discharges to the plant wastewater treatment system.

b. Venturi Scrubber

Cooled exhaust gases from the quench tower are directed to a variable-throat venturi scrubber which operates at a pressure differential of 15 to 30 inches of water. Water application rates are typically between 200 and 275 gpm, composed of secondary treated wastewater. Venturi effluents combine with the discharge of the demister tower. This heated water is directed to a portion of the plant wastewater system in which phenolic wastes are pretreated prior to mixing with general plant wastewaters.

c. Demister Tower

A water flow of 700 to 1000 gpm of secondary treated wastewater is maintained through the 12-foot-diameter demister.

d. Wet Electrostatic Precipitator

Exhaust gases from the demister are routed through an induced-draft fan into a single-field wet electrostatic precipitator. The emitting anodes and collection plates are arranged concentrically, and are cleaned with a continuous stream of water taken from the company's river intake, at a volume of 160 to 200 gpm. Effluent waters are directed to the general wastewater treatment system.

e. Stack

A 200-foot-tall, 12-foot-diameter brick stack vents emissions from the incinerator. As the gas discharge is usually saturated with water, the stack has a drain to the general wastewater treatment system, for which Dow Chemical estimates a flow of 1 gpm.

3. Other Waste Discharges

The types and volumes of effluent wastewaters from the air pollution control devices associated with the incinerator were described above. In addition, bulk solids falling from the rotary kiln are quenched and cooled in an ash trough through which a flow of 40 to 60 gpm of water drawn from the plant's Tittabawassee River intake is maintained. In addition, a small stream of untreated river water, estimated at 10 gpm, flows in a sluice under the rotary kiln's head end to receive and convey spills of heavy particulate matter falling from gaps in the seal. The effluent waters from the ash trough are routed to the general plant wastewater treatment system. About 15 to 20 cubic yards per day of solids are cleaned from the ash trough, and deposited in Dow Chemical's landfill located on Salzburg Road.

B. Sampling Strategy

1. Background

As indicated previously, the draft project plan for the National Dioxin Study Tier 4 - Combustion Sources formed the basis for selecting the input and discharge streams to be sampled in this study. However, that sampling scheme was formulated for a comparatively simple facility with a single waste input and a single air pollution control device. To adapt the model to the Dow Chemical incinerator, with two precombustion air intakes, four liquid waste feeds, and water effluent discharges from four air pollution control devices, required the collection of a significant number of samples to assess conditions during the emissions tests, and to evaluate compounds present in exhaust gases as they related to wastes incinerated on the test days.

2. Target Compounds

Table I-1 presents a list of samples and analyses required for a Tier 4 sampling program. The lists of target compounds presented in Tables V-1 and V-2 for air components, water, and solid samples build upon the primary list. In the case of the latter, the compounds added include several which may be precursors to formation of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) when subjected to elevated temperatures. These compounds were also selected based upon information about Dow Chemical manufacturing processes and liquid wastes consumed at the incinerator. For air components, compounds with known or suspected health effects when respired in ambient air, such as carbon tetrachloride and trichloroethylene, were added for the purpose of determining whether significant air emissions, from a public health standpoint, were occurring from the incinerator.

The compounds listed in Tables V-1 and V-2 are arranged by analysis type (PCDD/PCDF, semi-volatile organic (semi-VOA), and volatile organic (VOA)) to enable association with the sampling methods presented in the following section.

The Tier 4 sampling and analysis matrix shown in Table I-1 includes analyses for total organic chlorine in the two classes of input streams, and for PCDDs. It was determined that available methods for sampling for total organic chlorine

TABLE V-1

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
TARGET COMPOUNDS IN AIR

Analysis Type	
PCDD/PCDF	2378-TCDD
"	21 TCDD isomers
"	Total PeCDD
"	Total HxCDD
"	Total HpCDD
"	OCDD
"	2378-TCDF
"	Total TCDF
"	Total PeCDF
"	Total HxCDF
"	Total HpCDF
"	OCDF
Semi-volatile	Polychlorinated biphenyls (as positional isomer classes)
"	Other chlorinated biphenyls
"	Chlorinated phenols
"	Chlorinated benzenes
"	Diphenyl oxide
"	Chlorinated diphenyl oxides
"	Phenol
"	Phenyl phenol
"	Biphenyl
Volatile	Carbon tetrachloride
"	Ethylene dichloride
"	Perchloroethylene
"	Trichloroethylene
"	Vinylidene chloride

TABLE V-2

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR TARGET COMPOUNDS IN WATER AND SOLIDS DISCHARGES

Analysis Type	
PCDD/PCDF	2378-TCDD
"	21 TCDD isomers
"	Total PeCDD
"	Total HxCDD
"	Total HpCDD
"	OCDD
"	2378-TCDF
"	Total TCDF
"	Total PeCDF
"	Total HxCDF
"	Total HpCDF
"	OCDF
Volatile	Benzene
Semi-volatile	Biphenyl
Semi-volatile	Chlorinated biphenyls (as positional isomer classes)
Semi-volatile	Biphenyl phenyl ether
Volatile	Carbon tetrachloride
Semi-volatile	Chlorinated benzenes
Semi-volatile	Chlorinated phenols
Semi-volatile	Diphenyl oxide
Semi-volatile	Chlorinated diphenyl oxides
Semi-volatile	Divinyl benzene
Semi-volatile	Ethylbenzene
Volatile	Ethylene dichloride
Semi-volatile	Hydroxybenzaldehyde
Semi-volatile	Hydroxybenzoic acid (ortho and para)
Volatile	Perchloroethylene
Semi-volatile	Phenol
Semi-volatile	Phenyl phenols (ortho and para)
Volatile	Styrene
Semi-volatile	2,4,5-T and esters
Volatile	Trichloroethylene
Semi-volatile	Vinyl toluene (ethenyl methyl benzene)
Volatile	Vinylidene chloride
Volatile	Xylene

in liquid streams would not distinguish between organic and inorganic chlorine; also, the analytical methods were not of sufficient accuracy to be of use in this study. For this study, analyses for PCDDs and PCDFs were limited to the tetra-CDD isomers, total tetra- through hepta-CDD and CDF homologues, and OCDD and OCDF.

In addition to the above analytical work, incinerator emissions were evaluated with respect to requirements for hazardous waste incinerators developed pursuant to the Resource Conservation and Recovery Act (RCRA) and listed at 40 CFR Part 264.343(c). Specifically, particulate emissions for incinerators consuming hazardous waste are limited to 0.08 grain/dscf. In accordance with the regulation, the weight of particulate matter caught in the filter and probe wash portion of the Modified Method 5 train on each sampling day was reported and compared against the standard. Consistent with the requirements of EPA Method 5, this analysis did not consider any of the particulate matter trapped in the "back half" (impinger catch) of the train; in any event, such an analysis would have disrupted the determination of PCDDs and PCDFs in this portion of the train, and would likely have added little to the total catch of particulate matter.

3. Sampling Locations

The following streams and locations were selected for sampling based upon the unique characteristics of the Building 703 incinerator. These locations are also described in the quality assurance project plan written by the sampling contractor, GCA Corporation, in preparation for conducting sampling work.[5]

a. Precombustion Air

The Tier 4 project plan requires sampling of precombustion air if a significant source of suspected PCDD precursors is in the vicinity of the sampling site. It was known that 2,4,5-trichlorophenoxyacetic acid and other precursor chemicals were manufactured or handled at several locations within the Dow Chemical facility. Also, previous work by EPA and Dow Chemical established that surficial soils in the plant were contaminated with 2378-TCDD and other PCDDs, notably in the immediate area of the incinerator. For these reasons, precombustion air was sampled.

As indicated earlier, the rotary kiln and afterburner sections each have combustion air intakes. Given the proximity of these air intakes, it was estimated that ambient air quality would be similar at either intake; therefore, only one of the intakes was sampled. Owing to greater accessibility, the rotary kiln air intake was selected.

b. Liquid Waste Feeds

Each of the three liquid waste feed nozzles, and the low-BTU liquid waste nozzle, was fitted with an existing valve and spigot for sample collection. Samples were taken directly into holding containers from these spigots.

c. Incinerator Exhaust

Because a variety of sampling equipment was employed in collecting and analyzing exhaust gas samples, three separate locations were selected. As shown in Figure V-2, the sampling locations were between the outlet of the wet electrostatic precipitator and the stack breeching.

(1) PCDDs, PCDFs, and Semi-Volatile Compounds

An existing pair of four-inch sampling ports spaced 90° apart was used to operate two Modified Method 5 trains, one dedicated to PCDDs and PCDFs and the other to semi-volatile pollutants. The trains were operated simultaneously in the two ports, such that the probes did not interfere or cause turbulence with respect to each other. As Figure V-2 indicates, the ports were sufficiently separated from upstream and downstream bends in the three-foot-diameter exhaust duct, to comply with the requirements of EPA test Method 1.

(2) Volatile Compounds

A single four-inch-diameter sample port located about six feet downstream of a 90° duct bend was employed for gas sampling utilizing a Volatile Organic Sampling Train (VOST), described later in this report.

(3) Continuous Emissions Monitoring and Sampling for Vinylidene Chloride

Continuous emissions monitor (CEM) probes for oxygen, carbon monoxide, and carbon dioxide were inserted into the exhaust duct at a point about five feet downstream of the VOST, and three feet upstream of the stack breeching. In addition, several samples for direct analysis of vinylidene chloride were drawn through a separate probe inserted at this point.

The exhaust of a carbon-bed adsorbing column is located between the first and second sampling locations described above. The column is used to filter airborne hydrocarbon emissions displaced from the loading of liquid wastes into the incinerator tank farm. These emissions were formerly vented to the atmosphere. The adsorber was installed in July 1984 and operates only when liquid transfer to the tank farm occurs. As no other appropriate sampling locations were available for the VOST, CEM, and Tedlar bag samplers, it was decided to use those ports. The compounds detected in the exhaust gas were to be evaluated with respect to the likelihood they may have arisen from carbon bed operation, as well as combustion within the incinerator.

(4) Sampling Incinerator Emissions Before Control Devices

The Tier 4 sampling and analysis protocol (see Table I-1) prescribes that samples be taken for PCDDs and PCDFs at a point preceding air pollution control devices. The feasibility of obtaining these samples was evaluated early in the test planning process; no access to exhaust gases was available prior to the quench tower, other than a single port at the rear of the

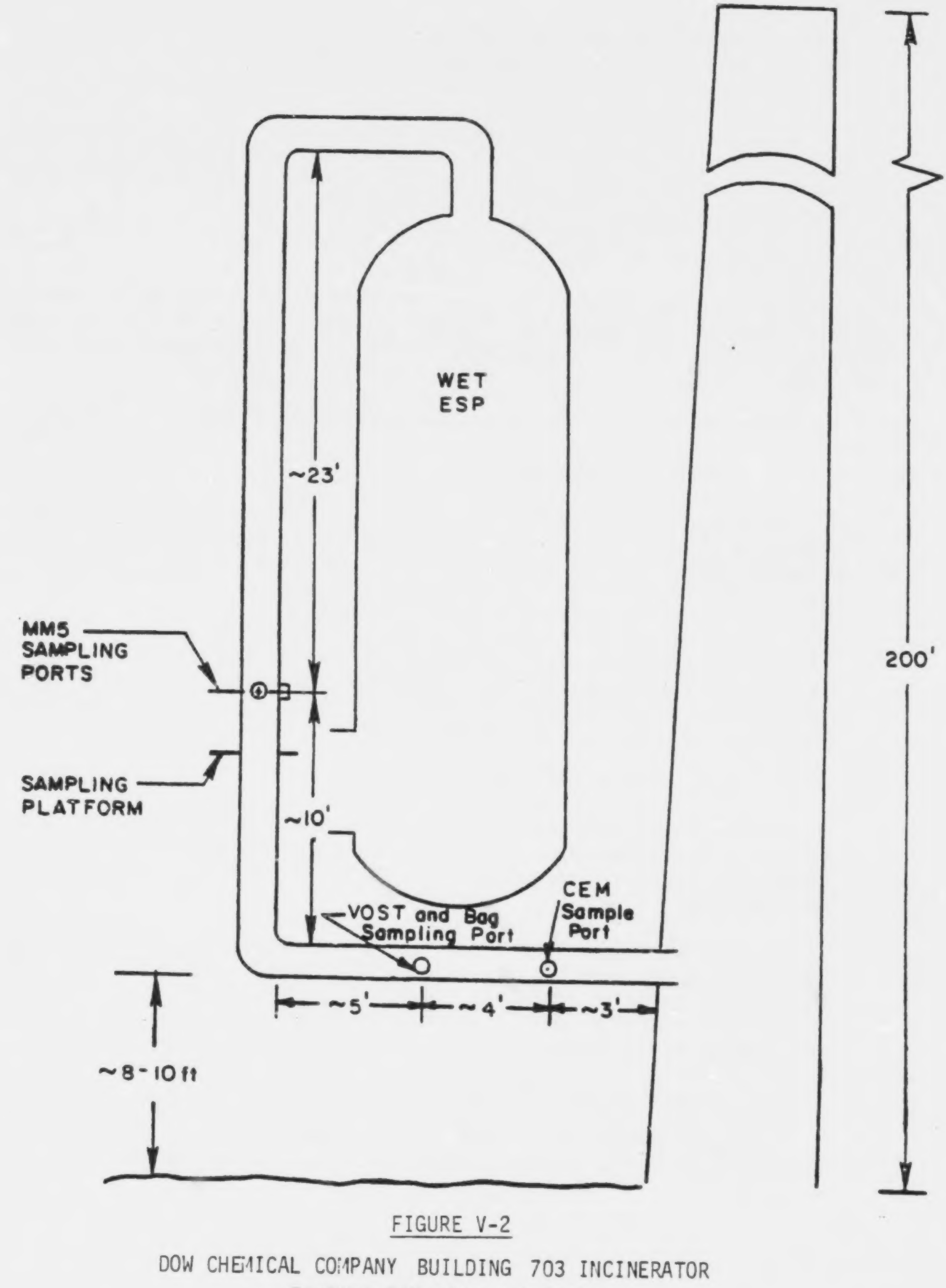

WET
ESP
~23'
MM5
SAMPLING
PORTS
SAMPLING
PLATFORM
~10'
VOST and Bag
Sampling Port
CEM
Sample
Port
~5'
~4'
~3'
~8-10 ft
200'

FIGURE V-2

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
EXHAUST GAS SAMPLING POINTS

incinerator afterburner which formerly held temperature monitoring probes. Because this location did not satisfy the requirements of EPA Method 1 in terms of separation from flow disturbances, it was rejected.

A second possible test location at the outlet of the quench tower was considered and rejected as only a single port was provided in a duct bend. Samples of exhaust gases prior to air pollution control devices were thus not taken as appropriate test locations were not available.

d. Incinerator Ash

Heavy solids are removed from the incinerator ash trough by a dragout chain which is operated for about ten minutes per hour. Grab samples were taken of material conveyed out of the trough by the scraper mechanism associated with the dragout chain.

e. Influent and Effluent Water and Control Device Ash

(1) Influent Service Water

Samples of the secondary treatment water returned to the incinerator to serve the quench tower, venturi, and demister were drawn from a one-inch-diameter spigot at the influent side of the quench tower. Suspended solids are filtered from this influent water immediately prior to the sampling point; therefore, separate samples or analyses of the solids portion of this stream were not specified. Samples of influent water from the Dow Chemical Tittabawassee River intake, which is directed to the ash trough and electrostatic precipitator, were not obtained. Previous sampling by EPA in 1981 showed PCDDs and PCDFs in this stream were not present at detection levels of 7 to 60 parts per quadrillion.

(2) Quench Tower Effluent

As described previously, waters leave the quench tower through two discharges. The composition of both discharges was expected to be similar; therefore, one discharge point, referred to as the west discharge, was chosen for sampling.

Advance inspections of the facility indicated the effluent streams from the quench tower were heavily laden with suspended solids from contact with incinerator exhaust gases. As the draft Tier 4 project plan specified that air pollution control device ash was to be analyzed separately, analyses of the aqueous and filterable solids portions of these samples were performed.

(3) Venturi Scrubber/Demister Effluent

Effluent streams from these devices combine prior to discharge to a sump located beneath the venturi scrubber, and samples were taken here. Analyses of aqueous and filterable solids portions were specified.

(4) Electrostatic Precipitator Effluent

These samples were obtained from a discharge sump beneath the electrostatic precipitator, and separate analyses of aqueous and filterable solids portions were requested.

(5) Ash Trough Water Effluent

About 50 gpm of water overflows the ash trough for discharge to the general wastewater treatment system. Samples were taken of this stream from a short open discharge flume located near the rotary kiln incinerator. The aqueous and filterable solids portions of each sample were analyzed separately.

C. Conduct of Study

Incinerator sampling was planned to include three separate days of operation, with each day's sampling spanning eight hours. This sampling period was chosen to assure that sufficient materials would be collected to enable compound detection, possible replicate analyses, and splitting of sample extracts between analytical laboratories.

The following influent and effluent streams of the Building 703 incinerator were sampled, when applicable, during the three-day study period:

Influent Streams

Precombustion Air (incinerator makeup)
Liquid Waste Feeds (maximum of four inlet nozzles)
Influent (service) Water

Effluent Streams

Incinerator Exhaust
Incinerator Ash
Effluent Water (from incinerator and air pollution control devices)
Air Pollution Control Device Ash

Each stream was sampled for the presence of PCDDs and PCDFs and the semi-volatile and volatile compounds referenced in Tables V-1 and V-2. In addition, the incinerator exhaust was sampled using a specialized analytical method specific to vinylidene chloride. Detailed information with respect to the conduct of the study, including sample handling and custody, analytical procedures, and incinerator operations while samples were obtained, appears in Appendix A of this report.

Sample collection and other field work were documented by the USEPA's field contractor (GCA Corporation) and USEPA personnel. Sample documentation included unique identification numbers and tags or labels; field workbooks; USEPA and GCA-generated sample custody records; and USEPA Sample Management Office Traffic Reports and Packing Lists.

Samples were gathered and handled according to the protocols outlined in the USEPA draft study plan[7] with minor revisions prompted by field conditions. Generally, the samples were composited over time to represent the entire sampling period on each day. However, for volatile compounds in aqueous streams (influent and effluent water) and in liquid waste feeds, single grab samples were taken as no reliable method was available to composite such samples. In addition, as discussed in the "Analytical Procedures" section of Appendix A, separate analyses were provided of the concentrations of the above constituents in the liquid and in the suspended or settleable solids (filtrate) portions of the influent and effluent waters. For volatile compound analyses, however, unfiltered grab samples of influent and effluent waters were analyzed as a whole.

The samples collected during this study were identified, packed (cooled as appropriate), and either shipped via commercial services for next-day arrival at contract laboratories, or, in the case of certain samples for which holding times prior to analysis were not of concern, delivered by USEPA personnel.

D. Analytical Procedures and Quality Assurance Reviews

1. Analytical Procedures

Selection of contract laboratories was coordinated by the USEPA Region V Central Regional Laboratory. Individual contracts were prepared for various groups of compound analyses, and sent to candidate laboratories for bid. The laboratories ultimately selected were the EAL Corporation, Richmond, California, for volatile compounds, semi-volatile compounds, pesticides, and PCBs; and the Brehm Laboratory, Wright State University, Dayton, Ohio, for PCDDs and PCDFs. For the former, analytical methods are detailed in Appendix A, Section III of this report. Analytical procedures for PCDDs and PCDFs are described in References 7 and 8, and Appendix C.

Several samples were taken of liquid wastes fed to the Building 703 incinerator. It was known in advance that these wastes were highly concentrated in single compounds, making them hazardous for analysis without prior extraction. Procedures for separating and aliquoting these waste samples are presented in Appendix B to this report. This work was carried out by Fred C. Hart Associates, Inc., Denver, Colorado. The extracts were shipped to EAL Corporation for analysis.

2. Quality Assurance Reviews

Data returned from the contract laboratories were reviewed for consistency with contract requirements by the USEPA Sample Management Office (Viar and Company, Alexandria, Virginia), and for adherence to quality assurance criteria contained in the Quality Assurance Project Plan developed for the study (see Reference 7) by the USEPA Region V Central Regional Laboratory. The results of these reviews are referenced in the discussion of general analytical findings which follows as Section V.F of this report.

E. Incinerator Operations During Tests

Extensive data concerning the operation of the Building 703 incinerator and the wastes burned during emissions testing were recorded by Dow Chemical, GCA Corporation, and USEPA personnel. This information is compiled in Appendix A, Section IV.

F. Results and Discussion

1. General Findings

Detailed results of sample analyses associated with the Building 703 incinerator emissions study are presented in Appendix D. Table V-3 summarizes the streams that were sampled and the compound groups analyzed. As indicated previously, no samples of loose or containerized solid wastes were taken owing to the unavailability of suitable sampling methods.

Detailed operating and sample analytical data were gathered during this study to enable association of emissions with such phenomena as incinerator temperatures, air pollution control device operations, wastes incinerated, and, if possible, to derive pollutant mass balances around the incinerator. However, as discussed in Section III.A. of Appendix D to this report, some limitations were placed on the acceptability of some of the data. For PCDDs and PCDFs, specifically, the recoveries of surrogate compounds used to assess the accuracy of analysis for certain homologue groups were not always within the relatively narrow ranges of acceptability established initially for this study. The ranges established in the study plan[7] were also revised to be consistent with those normally used by USEPA and others in the conduct of studies of PCDDs and PCDFs. In any event, overall completeness of PCDD and PCDF analytical data tended to be best for the tetra- through hexa-CDD homologues, for which health-related concerns are greatest.

For volatile and semi-volatile compounds, individual analyses were considered fully acceptable only if the recoveries of all introduced surrogate compounds were within prescribed ranges. There is no generally accepted protocol which would permit selective acceptance, compound by compound, based upon the recovery of specific single surrogates. However, for semi-volatile compound data, analytical results for an acid compound were considered valid if the recoveries of all acid surrogates were acceptable; the same was done for base-neutral compounds. Overall data completeness for semi-volatile compounds was based upon acceptable recoveries of all six surrogate compounds.

a. Influent Streams

(1) Precombustion Air

As described previously, this stream was sampled at the air intake of the rotary kiln portion of the Building 703 incinerator, but is taken to represent the characteristics of all air drawn into the incinerator and afterburner at any point preceding the combustion process. These data may also be used to assess the characteristics of ambient air in the immediate vicinity of the incinerator.

TABLE V-3

SAMPLING AND ANALYSIS SUMMARY
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR EMISSIONS STUDY
8/28, 8/30, 9/5/84

	Volatile Compounds[1]	Semi-Volatile Compounds[2]	Pesticides and PCB	PCDDs and PCDFs	Vinylidene Chloride
Influent Streams					
Precombustion Air	X	X		X	
Liquid Waste Feeds	X	X	X	X	
Influent (service) Water	X	X		X	
Effluent Streams					
Incinerator Exhaust	X	X		X	X
Incinerator Ash		X		X	
Effluent Water	X	X		X	
Air Pollution Control Device Ash[3]		X		X	

[1] Boiling points less than 100°C.
[2] Boiling points greater than 100°C.
[3] Analysis of filterable solids from effluent wastewater streams from individual air pollution devices associated with the incinerator.

Data for volatile pollutants (see Table V-4) showed the possible presence of the following compounds in the 1 to 100 parts-per-billion range:

carbon tetrachloride,
monochlorobenzene,
trichloroethylene, and
ethylbenzene.

However, the stringent quality assurance criteria (see discussion in Section III.A. of Appendix D) established for accuracy were such that only three of eight (38%) of the precombustion air volatile compound analyses were considered acceptable. Also, field duplicate sample analyses, limited to one of the three sampling days, suggested the quantitation of only monochlorobenzene to have been reliably established. The semi-volatile compound 1,4-dichlorobenzene was detected with good precision, and these data are included in Table V-4 for informational purposes; however, as no calibration standard for dichlorobenzene was run in the volatile compound analysis, the analytical method for semi-volatile compounds is more appropriate for this compound. Reference is made to Table V-5, where the detected semi-volatile compounds are listed.

Of the semi-volatile compounds (Table V-5), the following were present in concentrations of approximately 1 ug/m^3:

1,2-dichlorobenzene,
1,3-dichlorobenzene,
1,4-dichlorobenzene,
1,2,4-trichlorobenzene, and
naphthalene.

In addition, monochlorobenzene was detected at levels apparently lower than those indicated by the sampling method for volatile compounds.[4] However, this is not considered a reliable quantitation, as a calibration standard was not run for this analyte. Tentative findings of low levels of other benzene-ring compounds are detailed in Appendix D, Table D-3. All of the eight samples taken over the three-day period were found to be accurate within acceptable limits; however, precision criteria were met only for 1,2-dichlorobenzene and 1,2,4-trichlorobenzene.

Levels of PCDD and PCDF (Table V-6) ranging from approximately 10 to over 800 pg/m^3 of various homologues were found on all three sampling days, and about 5 pg/m^3 of 2378-TCDD was detected on the second sampling day. The data appear to show the consistent presence of TCDD, OCDD, TCDF, and OCDF, along with scattered findings of other homologues. More detailed data concerning TCDD isomers appear in Appendix D.

(2) Liquid Waste Feeds

The concentrated liquid wastes incinerated on the three sampling days contained a wide variety of volatile compounds, fully shown in Appendix D, Table D-10 (and summarized in Table V-7), including several chlorinated and

TABLE V-4

VOLATILE COMPOUNDS - PRE-COMBUSTION AIR
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

COMPOUND CONCENTRATION (ug/m^3)[1]

	COMPOUNDS DETECTED				
SAMPLING DATE	Carbon tetrachloride	Monochlorobenzene	Trichloroethylene	1,4-dichlorobenzene[2]	Ethylbenzene
8/28/84	15.35	12.87	0.64	72.43	10.63
8/28/84 (Field Duplicate)	61.00	20.55	3.12	60.20	29.26
8/30/84	222.50	--	16.98	81.69	--
9/5/84	26.69	29.04	--	45.35	--
Precision (RPD) 8/28/84 Samples	120	46	132	18	93

[1] Sample Concentration Less Field Blank Concentration
[2] Compound Tentatively Identified

TABLE V-5

SEMI-VOLATILE COMPOUNDS - PRE-COMBUSTION AIR
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

COMPOUND CONCENTRATION (ug/m^3)[1]

	TARGETED COMPOUNDS						OTHER COMPOUNDS DETECTED									
SAMPLING DATE	1,2-dichlorobenzene	1,3-dichlorobenzene	1,4-dichlorobenzene	1,2,4-trichloro-benzene	Naphthalene	Biphenyl	Monochlorobenzene	2-methylnaphthalene	Ethylbenzene[2]	1-ethyl-2-methylbenzene[2]	1-(methylethyl)-benzene[2]	1,2-diethylbenzene[2]	1,3-diethylbenzene[2]	1,2-diethenylbenzene[2]	Diphenylether	
8/28/84	1.42	0.15	1.41	1.58	0.44	2.22[2]	3.08		2.19	2.41	1.92	2.78	1.26	0.96	4.74	
8/30/84	0.84	--	0.74	0.86	0.08	0.90	0.53		0.50							
8/30/84 (Field Duplicate)	1.03	--	--	1.19	0.64	--	1.84	0.25								
9/5/84	3.73	0.07	3.24	2.59	1.23	--	--	1.65								
Precision (RPD) 8/30/84 Samples	21	--	--	32	156		111									

[1] Sample Concentration Less Field Blank Concentration
[2] Compound Tentatively Identified

TABLE V-6

INCINERATOR PRECOMBUSTION AIR - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
8-28-84												
MODIFIED METHOD 5 TRAIN	ND (7.86)	58.21	ND (11.01)	ND (6.62)	ND (12.02)	216.60	ND (7.89)	391.22	ND (6.07)	ND (16.2)	ND (27.50)	21.18
FIELD DUPLICATE	ND (11.32)	ND (53.4)	ND (131)	ND (125)	ND (5.43)	335.14	ND (29.2)	628.02	ND (6.01)	ND (4.20)	ND (8.45)	ND (30.2)
FIELD BLANK	(Sample analysis not returned from laboratory.)											
8-30-84												
MODIFIED METHOD 5 TRAIN	5.16	17.99	ND (2.30)	10.39	235.10	802.08	12.93	12.93	12.50	14.23	108.48	113.67
FIELD BLANK	ND (0.77)	ND (0.41)	ND (2.17)	ND (3.67)	ND (4.51)	ND (11.85)	ND (1.11)	ND (1.20)	ND (1.96)	ND (3.28)	ND (5.20)	ND (13.93)
9-5-84												
MODIFIED METHOD 5 TRAIN	ND (1.48)	38.90	ND (0.94)	ND (1.46)	98.14	306.51	ND (1.74)	206.60	ND (1.45)	ND 1.42	37.43	30.95
FIELD BLANK	ND (0.55)	ND (0.35)	ND (0.40)	ND (0.85)	ND (2.15)	ND (4.83)	ND (0.39)	ND (0.29)	ND (0.37)	ND (0.33)	ND (3.08)	ND (4.21)

Note: Data expressed in pg/m^3.
Detection level indicated in parentheses.

TABLE V-7
QUANTITATED VOLATILE COMPOUNDS - LIQUID WASTE INPUTS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

	TARGET COMPOUNDS			OTHER CHLORINATED COMPOUNDS			BENZENE RING COMPOUNDS					OTHER COMPOUNDS			
	Monochlorobenzene	Carbon tetrachloride	Chloroform	Methylene chloride	Chloromethane	Tetrachloroethylene	Benzene	Ethylbenzene	Styrene	Toluene	Total xylenes	Acetone	2-Butanone	2-Hexanone	Bromomethane
REAGENT BLANK 1				11			131					393	1057		
REAGENT BLANK 2					144		192								
8/28/84															
Nozzle BA								1494				1478			
Nozzle BA Dilution	15300					7700		2050	350						
Nozzle BB #1								35600	15900	1700	2700				
Nozzle BB #2										260					470
Nozzle BB #2 Dilution				50			311					1700			
Nozzle C										2370		990			
Nozzle C RERUN										2110		950			
Field Blank															
8/30/84															
Nozzle BA									35500	1950	1850			3400	
Nozzle BA Field Blank															
Nozzle BB #1															
Nozzle BB #1 Field Duplicate															
Nozzle BB #2												65			
Nozzle BB #2 Field Duplicate						845		2890			2920	14			
Nozzle BB #2 Field Duplicate RERUN	17700				77200	8450		2850		43400					
Nozzle C															
Nozzle C Field Duplicate										210	1600	260		9530	
Nozzle C Field Duplicate RERUN	(SEE 1 BELOW)							44700	42400	2440				9580	
Nozzle C Field Blank															
9/5/84															
Nozzle BA	7490				128,500	9180		1290					1		
Nozzle BA Dilution	4340				137,200	4400				9920					
Nozzle BB					1804			1540	1573						
Nozzle BB Field Blank					173										
Nozzle C		446,270	2970	4136	2838					4620	176,405				
Nozzle C Dilution		283,000	3260	3400	2900			96320	230,400	4340		(SEE 1 BELOW)			
Nozzle C Field Blank															

NOTES:

1. Sample extracts were diluted, prior to analysis, five times or more. Surrogate recoveries were therefore out of acceptable ranges.
2. Data expressed in mg/kg.

benzene-ring compounds. A complete listing of tentatively identified volatile compounds appears in Appendix D, Table D-11. However, nine of the 28 individual analyses for volatile compounds were judged to be unacceptable in terms of accuracy (see Appendix D, Table D-10), as recoveries for all surrogate compounds were not within the ranges established for the study. For semi-volatile compounds (Table V-8), 15 of the 29 samples submitted were analyzed with satisfactory accuracy for all six surrogate compounds. (see Appendix D, Table D-12); some chlorinated phenols and other benzene-ring compounds were qualitatively detected on the first and second sampling day. A discussion of possible limitations on the use of these semi-volatile data may be found in Appendix D, Section III.D.2.

Pesticides in the low ppm range were tentatively found (see Table V-9) on scattered occasions, but accuracy data were not submitted by the analytical laboratory, and available precision data appear poor. No PCBs were found in any liquid waste; however, the detection limit requested of the analytical laboratory, 5 ppb, was not met, by at least three orders of magnitude.

When found, PCDDs and PCDFs appeared in liquid wastes fed to the incinerator through nozzles BB and C. The data in Table V-10 show generally good precision between duplicate samples from these nozzles on the second sampling day; accuracy goals were generally met for surrogate compounds (see Appendix D, Table D-17). Of the TCDD isomers, the 1368 and 1379 were most prevalent.

(3) Low-BTU Liquid Waste

A comparison of volatile compounds detected (Table V-11) reveals, as expected, concentrations significantly lower in low-BTU liquid wastes than in the liquid wastes described above. Concentrations in the range of 10 to 100 ug/L were established on the third sampling day for vinylidene chloride, ethylene dichloride, and chloroform, within satisfactory limits for precision and accuracy. Of the semi-volatile compounds (Table V-12) only 1,2-dichlorobenzene was found, within acceptable quality assurance limits.

Analyses for PCDDs and PCDFs were generally successful in achieving accuracy goals for PCDD surrogates but not for PCDF (see Appendix D, Table D-17). Therefore, the TCDF data contained in Table V-13 may be suspect. However, homologue-by-homologue precision on the third sampling day met the goal established for the study (<50% relative percent difference). Dioxin homologues were limited to tetra, hepta, and octa, and low-ppq concentrations of TCDF were indicated. As with the liquid wastes described previously, most TCDD was composed of the 1368 and 1379 isomers; no 2378-TCDD was found.

TABLE V-8
QUANTITATED SEMI-VOLATILE COMPOUNDS - LIQUID WASTE INPUTS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

	TARGET COMPOUNDS				BENZENE RING COMPOUNDS				OTHER
	1,2-dichlorobenzene	Phenol	2,4,5-trichlorophenol	2,4,6-trichlorophenol	Naphthalene	2-methylnaphthalene	Anthracene	Fluorene	Diethylphthalate
REAGENT BLANK 1									
REAGENT BLANK 2									
8/28/84									
Nozzle BA									
Nozzle BA, 5X Dilution									
Nozzle BA, 20X Dilution									
Nozzle BB #1			4690		144	77			
Nozzle BB #1, 10X Dilution			1900						10500
Nozzle BB #1, 20X Dilution									60800
Nozzle BB #2									
Nozzle BB #2, 10X Dilution									
Nozzle C									
Field Blank (Nozzles BA & BB)									
8/30/84									
Nozzle BA						33			1390
Nozzle BA Field Blank				110					1130
Nozzle BB #1									
Nozzle BB #1 Field Duplicate	1406			8320	680	2320		190	
Nozzle BB #1 Field Duplicate, 5X Dilution	1570			5570		1950	560		
Nozzle BB #2									5930
Nozzle BB #2 Field Duplicate									110
Nozzle BB #2 Field Duplicate, 10X Dilution									
Nozzle C	1240		4450	4490	345		40	145	
Nozzle C Field Duplicate					62	27			
Nozzle C Field Duplicate, 10X Dilution									270
Nozzle C Field Blank									
9/5/84									
Nozzle BA									
Nozzle BA, 10X Dilution									
Nozzle BB									
Nozzle BB Field Blank									
Nozzle C									
Nozzle C, 10X Dilution									
Nozzle C Field Blank									

NOTE: Data expressed in mg/kg.

TABLE V-9
LIQUID WASTE INPUTS - QUANTITATED PESTICIDE/PCB COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, and 9/5/84

	PESTICIDES											PCB (AROCLORS)						
	Aldrin	Alpha - BHC	Beta - BHC	Gamma - BHC (Lindane)	Chlordane	4,4'-DDD	4,4'-DDT	Dieldrin	Endosulfan II	Heptachlor	Toxaphene	1016	1221	1232	1242	1248	1254	1260
REAGENT BLANK 1																		
REAGENT BLANK 2																		
8/28/84																		
Nozzle BA				0.3														
Nozzle BB #1				0.3														
Nozzle BB #2										0.1								
Nozzle C																		
Nozzles BA & BB Field Blank																		
8/30/84																		
Nozzle BA	1.4	7.5						2.5										
Nozzle BA Field Blank																		
Nozzle BB #1																		
Nozzle BB #1 Field Duplicate						0.4	0.6		0.4									
Nozzle BB #2																		
Nozzle BB #2 Field Duplicate				0.8														
Nozzle C																		
Nozzle C Field Duplicate	3.1	11.7				1.2		3.1	1.4	1.2								
Nozzle C Field Blank																		
9/5/84																		
Nozzle BA			0.2	0.1														
Nozzle BB																		
Nozzle BB Field Blank																		
Nozzle C							0.3											
Nozzle C Field Blank																		

NOTE: Data expressed in mg/kg. Where data are not stated, compound was not detected.

TABLE V-10
LIQUID WASTE INPUTS - QUANTITATED PCDD/PCDF
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, and 9/5/84

	2378 - TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378 - TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
REAGENT BLANK 1												
REAGENT BLANK 2												
8/28/84												
Nozzle BA												
Nozzle BA Field Blank												
Nozzle BB #1		5.8	11.8	1.2	2.8	22.0		9.2	0.8			1.2
Nozzle BB #2		0.5						0.4				
Nozzle C												
8/30/84												
Nozzle BA												
Nozzle BA Field Blank												
Nozzle BB #1		33.0	6.3	0.9	3.0	11.5	0.3	37.0	1.8	0.7	0.6	0.6
Nozzle BB #1 Field Duplicate		30.7	4.9	0.4	2.6	12.1	1.4	32.3	5.3			
Nozzle BB #2												
Nozzle BB #2 Field Duplicate												
Nozzle C		60.3	3.5	2.6	3.8	19.8		36.6	1.5	3.5	8.1	7.4
Nozzle C Field Duplicate		21.8	6.1	4.2	5.7	19.8	2.1	18.0	4.3	7.1	8.2	7.7
Nozzle C Field Blank												
9/5/84												
Nozzle BA												
Nozzle BB		5.9	0.8			1.2	0.2	6.5	0.2			
Nozzle BA Field Blank			(SAMPLE ANALYSIS NOT RETURNED FROM LABORATORY)									
Nozzle C		0.8						0.2				
Nozzle C Field Blank												

NOTES: Data expressed in ng/g. Where data are not stated, homologue was not detected.

TABLE V-11

LOW-BTU LIQUID WASTE - VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

																TENTATIVELY IDENTIFIED COMPOUNDS		
	1,1,1 -trichloroethane	benzene	toluene	ethylbenzene	styrene	methylene chloride	acetone	vinylidene chloride	ethylene dichloride	chloroform	perchloroethylene	chlorobenzene	total xylenes	carbon tetrachloride	trichloroethene	hexamethylcyclo-trisiloxane	1-(methylethyl)-benezene	propylbenzene
8/28/84																		
COMPOSITE SAMPLE						6												
FIELD BLANK						170												
9/5/84																		
COMPOSITE SAMPLE						1127	1163	127	86	12	378	260	429				2791	1160
FIELD DUPLICATE		91				1241	1302	137	93	13			7659	2916	8		6222	
FIELD BLANK	24	3	4	14	12											200		

NOTE: Data expressed in ug/L.

TABLE V-12
LOW-BTU LIQUID WASTE-SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

	QUANTITATED COMPOUNDS					TENTATIVELY - IDENTIFIED COMPOUNDS									
	1,2-dichlorobenzene	2-methylnaphthalene	bis(2-ethylhexyl)phthalate	1,4-dichlorobenzene	2,4,6-trichlorophenol	2,4-dimethyl-2,3-heptadien -5-yne	propylbenzene	undecane	1,4-dihydro-1,4-methano- naphthalane	2-12-(2-butoxyethoxy) ethoxyl-ethanol	1-phenyl-1,2-ethanediol	1,4-dimethylbenzene	1-ethenyl-3-methylbenzene	1,3-benzenedicarboxylic acid, methylester	1,1-biphenyl-2-ol
8/28/84															
Composite Sample															
Field Blank															
9/5/84															
Composite Sample	121	174	49			13	6	29	62	52	1051				
Field Duplicate	95	809	96	313	167				1807			461	591	2398	4368
Field Blank															

Note: Data expressed in ug/L.

TABLE V-13
LOW-BTU LIQUID WASTE - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
8/28/84												
COMPOSITE SAMPLE				10.4								
FIELD BLANK												
8/30/84												
(NO SAMPLE TAKEN - Low-BTU liquid waste was not incinerated on this day)												
9/5/84												
COMPOSITE SAMPLE		29.3			181	753		33.9				
FIELD DUPLICATE		22.8			132	570		46.4				
FIELD BLANK												
PRECISION (RPD) - SAMPLE AND FIELD DUPLICATE		25			31	28		31				

NOTES: 1. All data expressed in pg/g.
2. Blank spaces denote homologue not detected. Detection limits ranged from 0.2 to 10.2 ppt for TCDD and TCDF, to 8.9 to 162 ppt for OCDD and OCDF.

b. Effluent Streams

(1) Incinerator Exhaust

Analyses for volatile compounds presented in Appendix D, Tables D-30, D-31, and D-32, should be evaluated in light of previous comments concerning the stringency of the accuracy criteria established for this study. Among the compounds appearing in incinerator exhaust gases were carbon tetrachloride, monochlorobenzene, 1,2-dichlorobenzene, 1,4-dichlorobenzene, perchloroethylene, and trichloroethylene. These data are summarized below:

Table V-14

Approximate Concentrations of Volatile Compounds in Incinerator Exhaust 8/28, 8/30, 9/5/84

	Concentration, ug/m^3		
	8/28/84	8/30/84	9/5/84
carbon tetrachloride	ND	0.03-0.59	ND
monochlorobenzene	0.09-0.13	0.01-0.47	0.06-0.09
1,2-dichlorobenzene	ND	0.04-3.81	ND
1,4-dichlorobenzene	ND	ND-0.52	ND
perchloroethylene	ND	0.01-0.07	0.01-0.04
trichloroethylene	ND	0.001-0.01	ND

ND = not detected in concentration higher than in field blanks.

Note that the two dichlorobenzenes detected by the volatile compound sampling method are considered semi-volatile compounds under the boiling point definition established previously in this report. With respect to semi-volatile compounds, only the following were detected on the second sampling day, August 30, 1984. No semi-volatile compounds were found on the other sampling days.

Table V-15

Approximate Concentration of Semi-Volatile Compounds in Incinerator Exhaust 8/30/84

Compound	Concentration, ug/m^3
1,2-dichlorobenzene	115
1,4-dichlorobenzene	102
tetrachlorobenzene	25
naphthalene	33

However, as shown in Appendix D, Table D-36, these compounds were detected in the XAD-2 cartridge portion of the Modified Method 5 train, for which the recoveries of the three acid surrogates did not meet the accuracy goal of 20 to 180% established for the study (see Appendix D, Section III.A.). The above data should be evaluated in this context.

Table V-16 is a presentation of PCDD and PCDF emissions from the Building 703 incinerator, expressed in ng/m^3. These data were developed by summing the amounts of PCDD and PCDF found in each of the four components of the Modified Method 5 train. No 2378-TCDD was found, at detection limits of 0.02 to 2 ng/m^3.

The data presented in Table V-16A are expressed in units of ng/dscm, adjusted to standard temperature and pressure, and normalized to a 3% oxygen content in exhaust gas. This was done to render the data directly comparable to information presented in the draft Project Summary Report - National Dioxin Study Tier 4 - Combustion Sources (document EPA-450/4-84-014g, April 1986), in which emissions data for a wide range of sources are presented.

Vinylidene chloride was detected in exhaust gas at concentrations ranging between 28.1 and 279.8 ppb, as shown in Table V-17.

Prior to analysis, the Modified Method 5 trains used to sample for PCDDs and PCDFs were disassembled and the filter and probe wash portions were dried and weighed in a manner conforming to EPA Method 5. The particulate emissions of the incinerator on the three test days were found to be 0.0842, 0.0615, and 0.0784 grain/dscf. The arithmetic average of these data is 0.0747 grain/dscf. The Resource Conservation and Recovery Act standard for hazardous waste incinerators, appearing at 40 CFR Part 264.343(c), is 0.08 grain/dscf. That standard does not provide for arithmetic averaging to determine compliance.

To obtain the weights of filter and probe wash residues, these fractions were desiccated to constant weight. Because of possible losses from volatilization of PCDDs and PCDFs from the filters and probe washes, the data presented in Tables V-16 and V-16A may be biased low. However, low volatilities of PCDDs and PCDFs suggest any losses would not be significant.

(2) Incinerator Ash

Table V-18 includes the results of analyses for semi-volatile compounds present in incinerator ash sampled on the three test days.

Table V-19 shows the concentrations of PCDD and PCDF found in this ash. Among the PCDDs, the higher-chlorinated homologues were predominant, at low parts per billion levels. No 2378-TCDD was detected at the low parts per trillion range; other isomers, primarily the 1368, 1379, 1237 and 1238, were present at levels of about 0.1 to 1.2 ng/g. Of the PCDFs, the tetra, hepta, and octa homologues were found at low parts per billion concentrations.

TABLE V-16
INCINERATOR EXHAUST - PCDD/PCDF ANALYSES
EXPRESSED IN TERMS OF CONCENTRATION IN AIR (ng/m^3)
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
Modified Method 5 Train Catches												
8/28/84		[45.95]	6.49	0.88	0.21	0.93	1.51	[81.22]	[12.95]	[2.47]	0.26	0.06
8/30/84		43.75	1.94	0.37	0.84	2.52	1.67	76.98	4.28	1.95	0.55	0.17
9/5/84		4.92				0.47		124.8	0.07			

NOTES -

○ - Data out of control with respect to precision criteria (±50% RPD)

[ ] - Bracketed data denote homologues detected in filter and probe wash portion of Modified Method 5 train were deleted owing to unacceptable duplicate analysis results. Only a small fraction of total concentration detected was affected (see data in Appendix D, Table D-38).

- Matrix spike analyses indicated recoveries out of control for the following:
  - Filter and probe wash - PeCDD and HxCDF
  - XAD-2 cartridge - HpCDD and HpCDF

Other media in the sampling train showed acceptable matrix spike recoveries.

TABLE V-16A
INCINERATOR EXHAUST - PCDD/PCDF ANALYSES
CONCENTRATION EXPRESSED IN ng/dscm, ADJUSTED TO STANDARD
TEMPERATURE AND PRESSURE (68°F, 29.92 in.Hg), AND NORMALIZED TO 3% OXYGEN CONTENT
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
Modified Method 5 Train Catches												
8/28/84		[116.8]	16.49	◯2.24	0.53	2.36	3.84	[206.4]	[32.91]	[6.28]	0.66	0.15
8/30/84		123.8	5.49	1.05	2.38	7.13	4.72	217.8	12.11	5.52	1.56	0.48
9/5/84		11.37				1.09		288.5	0.17			

NOTES -

◯ - Data out of control with respect to precision criteria (±50% RPD)
[ ] - Bracketed data denote homologues detected in filter and probe wash portion of Modified Method 5 train were deleted owing to unacceptable duplicate analysis results. Only a small fraction of total concentration detected was affected (see data in Appendix D, Table D-38).
- Matrix spike analyses indicated recoveries out of control for the following:
  - Filter and probe wash - PeCDD and HxCDF
  - XAD-2 cartridge - HpCDD and HpCDF

Other media in the sampling train showed acceptable matrix spike recoveries.

TABLE V-17
RESULTS OF SAMPLING FOR VINYLIDENE CHLORIDE
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

DATE	SAMPLE RUN	SAMPLE COLLECTION TIME (EDT)	VINYLIDENE CHLORIDE CONCENTRATION (ppbv)	STANDARD DEVIATION
8/28/84	1	1230-1330	88.6 (83.1, 88.0, 94.7)	5.8
	2	1405-1510	68.3 (72.1, 72.3, 60.2)	6.9
	3	1525-1625	64.3 (113.0*, 67.5, 61.1)	4.5
	4	1640-1735	74.5 (73.9, 74.7, 74.8)	0.5
	5	1750-1845	88.9 (94.2, 88.4, 84.1)	5.1
	6	1850-1930	112.4 (113.6, 111.2, 138.6*)	1.7
	7	1935-2015	104.4 (102.1, 107.8, 103.3)	3.0
8/30/84	1	1000-1050	149.7 (150.0, 154.9, 144.3)	5.3
	2	1100-1200	187.6 (180.9, 189.3, 192.7)	6.1
	3	1210-1250	241.6 (263.7, 219.5, 402.7*)	31.3
	4	1300-1350	279.8 (275.3, 285.9, 278.3)	5.5
	5	1400-1450	218.0 (219.6, 216.3)	2.3
	6	1500-1550	28.1 (28.9, 27.9, 27.6)	0.7
9/5/84	1	1000-1045	88.7 (94.3, 93.3, 78.5)	8.8
	2	1100-1150	70.3 (69.4, 68.9, 72.6)	2.0
	2 DUPLICATE	1100-1150	79.3 (76.7, 81.9, 79.3)	2.6
	3	1200-1245	157.8 (156.4, 152.5, 164.4)	6.1
	4	1400-1445	154.3 (162.2, 143.5, 157.2)	9.7
	5	1500-1545	156.0 (154.7, 161.6, 151.8)	5.0
	6	1600-1630	143.5 (146.6, 143.3, 140.6)	3.0

* Rejected as greater than one standard deviation from mean of three analyses.

TABLE V-18
INCINERATOR ASH SEMI-VOLATILES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
(Results in ug/kg)

	1,2-dichlorobenzene	1,4-dichlorobenzene	1,2,4-trichlorobenzene	phenol	di-n-butyl phthalate	diethyl phthalate	bis(2-ethylhexyl) phthalate	diphenylether	sulfur	biphenyl	2-ethylbiphenyl	methyldiphenylsilane	1,1'-(1,2-ethendiyl)bis(z) benzene	1,1':2',1-terphenyl	1,1':3',1-terphenyl	1,1':4',1-terphenyl	octamethyltrisiloxane	2-phenoxy-1,1'-biphenyl	methylbiphenylsilane	bis (2-methylpropyl) phthalate
8/28/84					433			201	2722											
8/28/84 Field Blank																				
8/30/84					1933						7189	52,838	11,628	4932	10,792	11,243				
8/30/84 Field Dup.	520	460	867		1733			10,883				44,757	5661	9919	6245	9965	2006	3681		
8/30/84 Field Blank	(SAMPLE ANALYSIS NOT RETURNED FROM LABORATORY)																			
9/5/84				363	1110	423	530		170	435		321								1069
9/5/84 Field Blank	(SAMPLE ANALYSIS NOT RETURNED FROM LABORATORY)																			

TABLE V-19
INCINERATOR ASH - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, AND 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
8/28/84												
COMPOSITE SAMPLE	ND (27.7)	1170	ND (19.1)	793	6060	32,700	66	9160	68	455	1520	2570
FIELD BLANK	ND (8.2)	ND (9.6)	ND (35.8)	ND (17.5)	ND (12.7)	ND (25.8)	ND (12.6)	ND (12.8)	ND (21.2)	ND (19.6)	ND (15.9)	ND (23.4)
8/30/84												
COMPOSITE SAMPLE	ND (23.1)	131	ND (13.6)	129	806	3180	17	594	ND (5.4)	44	449	573
FIELD DUPLICATE	ND (11.8)	107	ND (15.6)	111	498	2370	ND (11.3)	263	ND (7.3)	37	248	399
PRECISION (RPD)		20		15	47	29		77		17	58	36
FIELD BLANK	ND (7.1)	ND (3.1)	ND (15.5)	ND (6.1)	ND (15.5)	ND (25.8)	ND (4.2)	ND (5.4)	ND (7.4)	ND (8.6)	ND (21.7)	ND (11.3)
9/5/84												
COMPOSITE SAMPLE	ND (6.9)	71	ND (16.2)	ND (10.9)	76	266	ND (6.5)	540	ND (7.8)	ND (19.5)	ND (20.2)	78
FIELD BLANK		(Analytical data not returned from laboratory)										

NOTE: Data expressed in pg/g.

(3) Aqueous Influents and Effluents

Chloroform and carbon tetrachloride were found at low parts per trillion levels in service water supplied to the incinerator air pollution control devices. Otherwise, there were no consistent findings of any other volatile compounds (see Appendix D, Tables D-50, D-51, and D-52). Other than scattered detection of phthalate compounds, few semi-volatile compounds were found in effluent wastewaters. On the second and third sampling days, various chlorophenols and chlorobenzenes were found in influent service water, and biphenyls and terphenyls appeared in effluent wastewaters. Any association between these compounds is speculative.

Tables V-20, V-21, and V-22 are detailed presentations of PCDD and PCDF data for influent and effluent waters. Of particular interest is the apparent strong affinity of PCDD and PCDF for the filterable solids present in these waters. Also, some TCDD, TCDF, HpCDF, and OCDF were detected in influent service waters. No 2378-TCDD was found at any time, at detection limits of approximately 1 ng/L for aqueous samples and 10 ng/g for solids samples. Detailed information with respect to the TCDD isomers detected appears in Appendix D, Tables D-60, D-61, and D-62.

2. Quality Assurance Review

As indicated in the Quality Assurance Project Plan for the incinerator exhaust study (Reference 7), a goal of 90% was established with respect to the completeness of the analytical data. This measure was devised to assess the overall suitability of groups of data; individual data points were judged to be complete if precision and accuracy criteria applicable to a particular type of sample were met. In retrospect, given the complexity of this study this goal was overly optimistic.

Field duplicate samples were obtained on one of the three study days for each sample type. Where calculable, precision data are presented in the data tables and discussion included in Section V and Appendix D of this report. Owing to the wide range of compounds sought for analysis in each sample, and the number of field duplicate samples taken, there were few cases in which the same compounds were found in both actual samples and the field duplicates. The quality assurance objective was ± 50% or ± the detection limit. Because of the wide variety of compounds detected and the few opportunities to assess precision, the following discussion centers on completeness based solely on data accuracy, as measured by analysis of surrogate compounds introduced to each sample by the laboratories during analysis. These data are presented in the raw data summary tables in Appendix D, and, where appropriate, in the data tables in Section V. Several cases surfaced in which surrogate compounds were not detected or recovered less than 10%. Laboratory personnel indicated these samples were generally diluted during analysis such that some of the surrogate peaks were lost.

Table V-23 is a summary of data completeness for the categories of samples and compound groups other than PCDDs and PCDFs analyzed in this study. Generally, the completeness goal of 90%, established in the plan for the study[7],

TABLE V-20
AQUEOUS INFLUENTS AND EFFLUENTS - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	Total OCDF
Service Water	ND (.0021)	0.0384	ND (.0043)	ND (.0086)	ND (.0073)	0.198	ND (.0011)	1.26	ND (.0026)	ND (.0057)	0.0558	ND (.0130)
Quench Water (Water)	ND (.0013)	ND (.0010)	ND (.0010)	ND (.0042)	ND (.0079)	ND (.0206)	ND (.0005)	0.0025	ND (.0015)	ND (.0029)	ND (.0055)	ND (.0118)
Quench Water (Solids)	ND (15.6)	432	54.9	43.7	274	1437	11.0	170	66.4	117	427	379
Venturi/Demister Water (Water)	ND (.0011)	ND (.0010)	ND (.0027)	ND (.0026)	ND (.0059)	ND (.0147)	ND (.0002)	0.0393	ND (.0022)	ND (.0018)	ND (.0030)	ND (.0139)
Venturi/Demister Water (Solids)	ND (2.98)	238	82.0	55.1	265	1113	8.52	137	100	130	337	284
ESP Water (Water)				SAMPLE ANALYSIS DATA NOT RETURNED FROM LABORATORY								
ESP Water (Solids)				SAMPLE ANALYSIS DATA NOT RETURNED FROM LABORATORY								
Ash Pit Water (Water)	ND (.0003)	ND (.0010)	ND (.0010)	ND (.0027)	ND (.0058)	ND (.0289)	ND (.0003)	ND (.0010)	ND (.0031)	ND (.0012)	ND (.0066)	ND (.0121)
Ash Pit Water (Solids)	ND (19.8)	ND (23.3)	ND (171)	ND (94.3)	ND (126)	323	ND (27.4)	189	ND (45.1)	ND (42.5)	ND (91.5)	ND (118)
Effluent Water Field Blank	ND (.0003)	ND (.0010)	ND (.0016)	ND (.0026)	ND (.0083)	ND (.0130)	ND (.0002)	ND (.0010)	ND (.0039)	ND (.0014)	ND (.0055)	ND (.0098)
Effluent Water Backup Field Blank	ND (.0002)	ND (.0010)	ND (.0054)	ND (.0115)	ND (.0275)	ND (.0447)	ND (.0003)	ND (.0010)	ND (.0037)	ND (.0075)	ND (.0167)	ND (.0284)

Note - Date expressed in ng/g for solids samples, ng/L for aqueous samples.

TABLE V-21
AQUEOUS INFLUENTS AND EFFLUENTS - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
Service Water	ND (.0027)	0.0464	ND (.0019)	ND (.0021)	0.0179	0.187	ND (.0012)	1.42	0.0088	ND (.0067)	0.0167	0.0477
Quench Water (Water)	ND (.0007)	ND (.0010)	ND (.0024)	ND (.0042)	ND (.0115)	ND (.0301)	ND (.0001)	0.0223	ND (.0037)	ND (.0028)	ND (.0131)	ND (.0168)
Quench Water (Solids)	ND (11.1)	707	99.3	75.3	460	2358	15.4	182	ND 87.5	124	785	641
ESP Water (Water)	ND (.0009)	.0062	ND (.0011)	ND (.0028)	ND (.0057)	ND (.0192)	ND (.0004)	0.287	ND (.0051)	ND (.0037)	ND (.0055)	ND (.0182)
ESP Water (Water) Field Duplicate	ND (.0028)	.0189	ND (.0019)	ND (.0029)	ND (.0044)	ND (.0077)	ND (.0004)	0.607	ND (.0039)	ND (.0017)	ND (.0070)	ND (.0099)
ESP Water (Solids)	ND (35.3)	4212	885	147	417	2199	45.3	539	405	75.7	150	200
ESP Water (Solids) Field Duplicate	ND (65.5)	1864	393	205	515	2530	47.7	6574	345	58.6	161	226
Venturi/Demister Water (Water)	ND (.0006)	ND (.0010)	ND (.0012)	ND (.0021)	ND (.0089)	ND (.0075)	ND (.0005)	0.0682	ND (.0021)	ND (.0033)	ND (.0056)	ND (.0164)
Venturi/Demister Water (Solids)	ND (2.08)	307	49.2	27.6	162	707	3.22	168	64.6	82.9	199	283
Ash Pit Water (Water)	ND (.0010)	ND (.0025)	ND (.0240)	ND (.0227)	ND (.0292)	ND (.0453)	ND (.0022)	ND (.0038)	ND (.0120)	ND (.0110)	ND (.0232)	ND (.0269)
Ash Pit Water (Solids)	ND (1.08)	15.9	ND (3.09)	ND (3.14)	21.5	94.9	ND (1.71)	114	ND (3.15)	ND (2.93)	10.0	12.5
Effluent Water Field Blank	ND (.0005)	ND (.0010)	ND (.0011)	ND (.0021)	ND (.0031)	ND (.0053)	ND (.0006)	ND (.0010)	ND (.0024)	ND (.0017)	ND (.0052)	ND (.0037)
Effluent Water Backup Field Blank	ND (.0005)	ND (.0010)	ND (.0080)	ND (.0063)	ND (.0083)	ND (.0104)	ND (.0014)	ND (.0025)	ND (.0077)	ND (.0128)	ND (.0046)	ND (.0127)

Note - Data expressed in ng/g for solids samples, ng/L for aqueous samples.

TABLE V-22
AQUEOUS INFLUENTS AND EFFLUENTS - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
Service Water	ND (0.341)	ND (0.229)	ND (0.556)	ND (0.720)	ND (0.318)	ND (0.520)	ND (0.192)	ND (0.517)	ND (0.299)	ND (0.351)	ND (0.627)	ND (0.396)
Quench Water (Water)	ND (.0004)	ND (.0010)	ND (.0024)	ND (.0027)	ND (.0018)	ND (.0020)	ND (.0001)	0.0058	ND (.0015)	ND (.0015)	ND (.0012)	ND (.0011)
Quench Water (Solids)	ND (1.10)	73.9	ND (7.43)	ND (3.19)	69.0	236	ND (1.93)	830	7.09	16.1	125	103
Venturi/Demister Water (Water)	ND (.0008)	ND (.0010)	ND (.0021)	ND (.0031)	ND (.0036)	ND (.0064)	ND (.0001)	0.0157	ND (.0010)	ND (.0024)	ND (.0017)	ND (.0035)
Venturi/Demister Water (Solids)	ND (1.29)	56.3	17.5	7.35	44.3	261	2.05	723	22.3	19.7	69.1	84.8
ESP Water (Water)	ND (.0014)	0.0052	ND (.0104)	ND (.0039)	ND (.0087)	ND (.0051)	ND (.0015)	0.0995	ND (.0041)	ND (.0030)	ND (.0026)	ND (.0061)
ESP Water (Solids)	ND (28.2)	247	61.5	20.3	96.0	423	9.70	90.0	47.0	14.7	68.2	82.1
Ash Pit Water (Water)	ND (.0003)	ND (.0010)	ND (.0012)	ND (.0017)	ND (.0029)	ND (.0025)	ND (.0001)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0021)	ND (.0037)
Ash Pit Water (Solids)				(SAMPLE ANALYSIS DATA NOT RETURNED FROM LABORATORY)								
Effluent Water Field Blank	ND (.0013)	ND (.0010)	ND (.0016)	ND (.0071)	ND (.0067)	ND (.0088)	ND (.0023)	ND (.0022)	ND (.0080)	ND (.0025)	ND (.0049)	ND (.0057)
Effluent Water Backup Field Blank	ND (.0003)	ND (.0010)	ND (.0048)	ND (.0027)	ND (.0039)	ND (.0058)	ND (.0002)	ND (.0010)	ND (.0025)	ND (.0027)	ND (.0026)	ND (.0039)

Note - Data expressed in ng/g for solids samples, ng/L for aqueous samples.

TABLE V-23
OVERALL DATA COMPLETENESS
BASED UPON ANALYTICAL ACCURACY CRITERIA
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR EMISSIONS STUDY

	Analysis Type			PCDD/PCDF Internal Standards			
Type of Sample	Volatile Compounds	Semi-Volatile Compounds	Pesticides/PCB	$^{13}C_{12}$-2378-TCDD	$^{37}Cl_4$-2378-TCDD	$^{13}C_{12}$-OCDD	$^{37}Cl_4$-2378-TCDF
Precombustion Air	38% (3/8)	88% (7/8)	--	71% (5/7)	86% (6/7)	29% (2/7)	71% (5/7)
Liquid Wastes	68% (19/28)	52% (15/29)	0% (0/19)[1]	84% (16/19)	95% (18/19)	74% (14/19)	84% (16/19)
Low-BTU Liquid Waste	100% (5/5)	80% (4/5)	--	80% (4/5)	100% (5/5)	100% (5/5)	20% (1/5)
Incinerator Exhaust	13% (2/16)	57% (16/28)	--	79% (19/24)	83% (20/24)	75% (18/24)	67% (16/24)
Incinerator Ash	--	71% (5/7)	--	86% (6/7)	86% (6/7)	86% (6/7)	86% (6/7)
Aqueous Influents and Effluents	95% (21/22)	89% (31/35)	--	74% (26/35)	91% (32/35)	74% (26/35)	60% (21/35)

[1] Owing to dilution effects during analysis, the target detection limit was not met.

was not met, but in many cases was nearly met. In any event, this performance should be evaluated with respect to comments made previously about the stringency of the accuracy criteria used to judge the acceptability of volatile and semi-volatile compound analyses.

Similar data for PCDDs and PCDFs may be found in the tables in Appendix D in which analytical results are presented. As indicated previously, accuracy with respect to the TCDD surrogates deemed most important in evaluating potential health risks, was generally near 80%; for OCDD and PCDFs, accuracy was less reliable, but these compounds are of less concern regarding health risk assessment.

The above-referenced study plan also describes desired detection limits for the types of samples and analytical procedures employed in this study. These data (Table V-24) indicate detection limits were met or nearly met for volatile and semi-volatile analyses except those of liquid wastes, where sample extraction and dilution was necessary. For PCDDs and PCDFs, requested detection limits were very low, but were met in several cases. Of particular interest is the demonstrated detection of PCDDs and PCDFs in incinerator exhaust in the XAD-2 sorbent portion of the Modified Method 5 train, where a significant portion of PCDDs and PCDFs was trapped.

3. Discussion of Results

The concentrations of PCDDs and PCDFs entering and discharged from the Building 703 incinerator on the three sampling days, are presented in detail in Appendix D, Tables D-64 through D-66; Tables D-67 through D-69 of that appendix show similar data for TCDD isomers. Detailed summaries of incoming and outgoing loadings of PCDDs and PCDFs, and TCDD isomers, are presented in Appendix D, Tables D-70 through D-75.

It must be remembered in interpreting these data that a major waste stream introduced to the incinerator, the loose and containerized solid wastes, could not be representatively sampled in this study. While no samples of Tittabawassee River water were taken (this being a component of some waters taken in and circulated through air pollution control devices), concentrations of PCDDs and PCDFs were expected to be either not present or not significant in this stream. Samples obtained by EPA as part of a 1981 water sampling study[9] support this conclusion. At that time, 2378-TCDD and other dioxin homologues were not found in the Tittabawassee River water intake to the Dow Chemical plant at detection levels in the parts per quadrillion range.

Total suspended solids (TSS) concentrations used to calculate discharged PCDD and PCDF loadings in the solid portions of the wastewater streams were taken from data developed by the analytical laboratory during analysis of PCDDs and PCDFs. These data are stated below, and compared with data for those streams gathered on four separate days in 1984 by Dow Chemical[20], and during a sampling program conducted on August 28-29, 1984, by the USEPA Region V Eastern District Office:

TABLE V-24
COMPARISON OF ACTUAL AND DESIRED DETECTION LIMITS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR EMISSIONS STUDY

	Volatile Compounds		Semi-Volatile Compounds		Pesticides/PCB			PCDD/PCDF	
	Detection Limit		Detection Limit		Detection Limit			Detection Limit	
Type of Sample	Desired	Actual	Desired	Actual	Desired	Actual		Desired	Actual
Precombustion Air	1 ppb	0.3-0.8 ppb	5 ppb	0.05 ppb		---	2378-TCDD 2378-TCDF Total TCDD Total TCDF Cl_5-Cl_8 CDD Cl_5-Cl_8 CDF	2 ng 2 ng 2 ng 2 ng 6 ng 6 ng	4.7-94 ng 3.3-242 ng 3-433 ng 2.4-9.3 ng 3.4-1038 ng 2.8-250 ng
Liquid Wastes	1 ppb	1 ppm	5 ppb	1-10 ppm	5 ppb	100 ppb	TCDD/TCDF OCDD/OCDF	30 ppq 90 ppq	0.25-10.6 ppt 0.77-40.6 ppt
Low-BTU Liquid Wastes	1 ppb	3 ppt	5 ppb	6 ppt		---	TCDD/TCDF OCDD/OCDF	30 ppq 90 ppq	14-714 ppq 230-7940 ppq
Incinerator Exhaust	1 ppb	0.25-0.50 ppb	5 ppb	1-2 ppb		---	Impingers XAD-2 sorbent	30-90 ppq 2-6 ng	~5-100 ppt 0.52-126 ng
Incinerator Ash		---	5 ppb	0.5 ppb		---	TCDD/TCDF Cl_5-Cl_8 PCDD/PCDF	5 ppt 15 ppt	0.5-1.9 ppt ~0.3-2.0 ppt
Aqueous Influents and Effluents	1 ppb	5 ppb	5 ppb	10 ppb		---	Water Solids	30-90 ppq 5-15 ppt	~20-1600 ppq ~60-6000 ppt

TOTAL SUSPENDED SOLIDS (mg/L)

Water Stream	EPA Incinerator Study 8/28/84	EPA Incinerator Study 8/30/84	EPA Incinerator Study 9/5/84	Dow Chemical (1984)	EPA 8/28-29/84
Quench Tower	71	111	127	106- 488	840
Venturi/Demister	77	132	169	72-1144	276
ESP	--	16	240	42- 444	34
Ash Pit	3	132	156	46- 393	82

These data illustrate the variability of TSS concentrations in the wastewater streams. Effluent loadings of PCDDs and PCDFs in incinerator ash were calculated based upon a density of 0.66 ton per cubic yard, as supplied by Dow Chemical[20], and a disposal rate, as described previously, of 15 to 20 cubic yards per day. Loadings of discharged PCDDs and PCDFs stated in the tables correspond to the range of 15 to 20 cubic yards of incinerator ash disposed daily (see Section V.A. of this report).

Three of the loadings tables (Appendix D, Tables D-70 through D-72) are averaged over the three days of sampling and summarized in Figure V-3 through V-6 for total TCDD, OCDD, TCDF, and OCDF. In general, the data presented in these figures indicate loadings in streams discharged from the Building 703 incinerator were comparable to or higher than in those fed to it. There appears to be a strong tendency for the higher chlorinated homologues (penta- and higher) to reside in the solid discharges, such as the effluent water solids portions and incinerator ash.

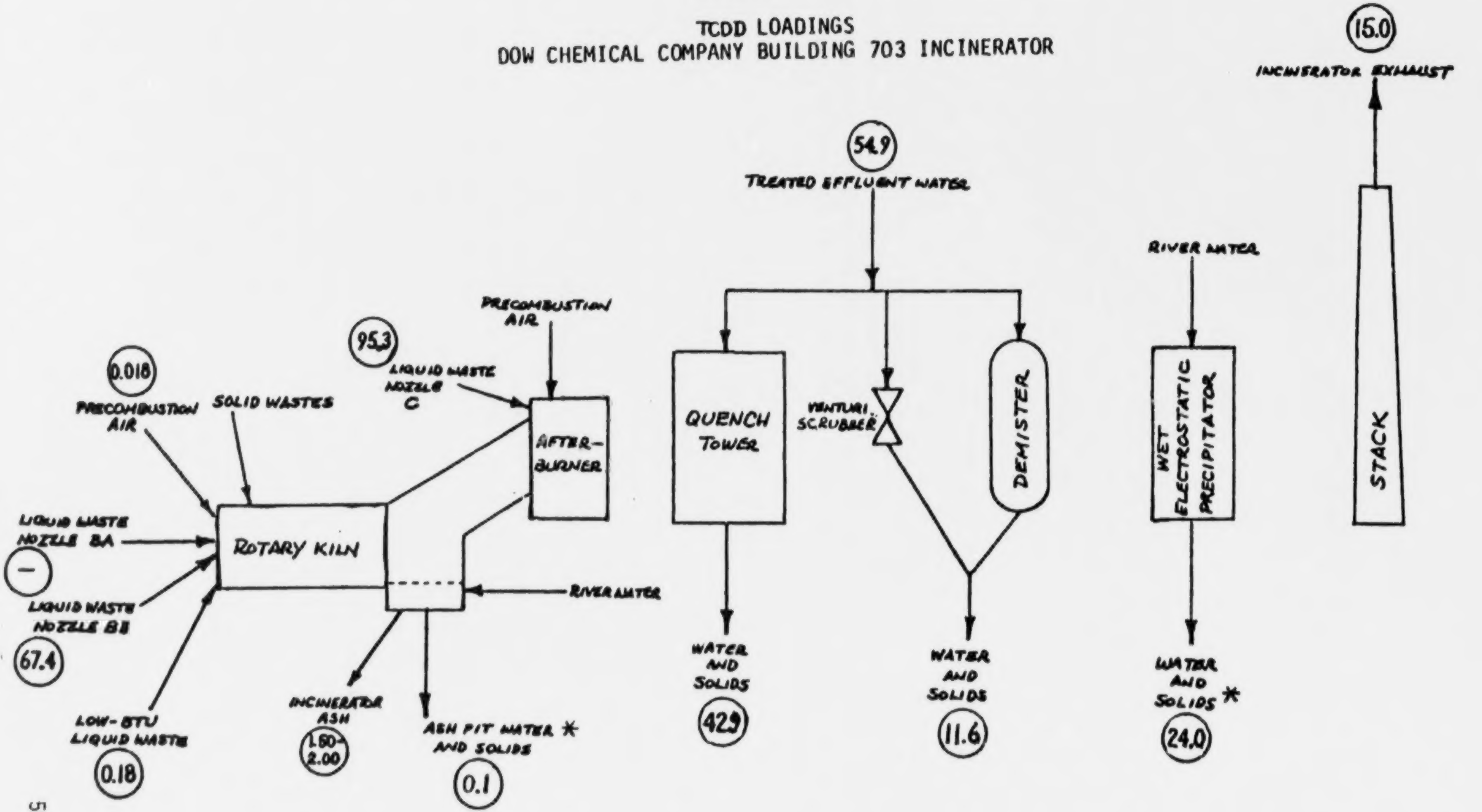
An objective of this study was to relate air, water, and solids emissions of PCDDs and PCDFs and other compounds from the Building 703 incinerator to the operational characteristics and waste materials consumed in the facility. As the operational characteristics (incinerator temperatures, air pollution control device water flows, exhaust gas oxygen content, etc.) appeared similar over the three sampling days, with the exception of an electrostatic precipitator arcing phenomenon described in Section IV.B.7 of Appendix A, it is thought the differences in PCDD and PCDF emissions appearing in Tables V-16, V-19, and V-20 through V-22 may have been attributable to waste content.

In exhaust gas, in general, the highest concentrations of penta- and hexa-CDD and CDF were found on the first sampling day, and of hepta- and octa-CDD and CDF on the second day. Similar concentrations of TCDD and TCDF were detected on the first and second days, with the lower concentration of TCDD and similar concentration of TCDF on the third day. In effluent wastewaters, highest concentrations of most homologues appeared on the second day.

Incinerated loose and containerized solid wastes were not defined sufficiently to discern any correlations in this area, and the liquid waste feed from nozzle "BA" was similar on all three days. It was established in the analytical results that the relative concentrations of most compounds in low-BTU liquid waste were lower than in any of the concentrated liquid wastes. While extensive data on incinerator operating temperatures, pressures, air pollution control device water, and flow rates were obtained (see Appendix A, Table A-3),

FIGURE V-3

TCDD LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


15.0
INCINERATOR EXHAUST
54.9
TREATED EFFLUENT WATER
RIVER WATER
PRECOMBUSTION AIR
95.3
LIQUID WASTE NOZZLE C
0.018
PRECOMBUSTION AIR
SOLID WASTES
AFTER-BURNER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WET ELECTROSTATIC PRECIPITATOR
STACK
LIQUID WASTE NOZZLE 8A
ROTARY KILN
—
RIVER WATER
LIQUID WASTE NOZZLE 8B
67.4
WATER AND SOLIDS
WATER AND SOLIDS
WATER AND SOLIDS *
LOW-BTU LIQUID WASTE
0.18
INCINERATOR ASH
1.80-2.00
ASH PIT WATER AND SOLIDS *
0.1
42.9
11.6
24.0

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

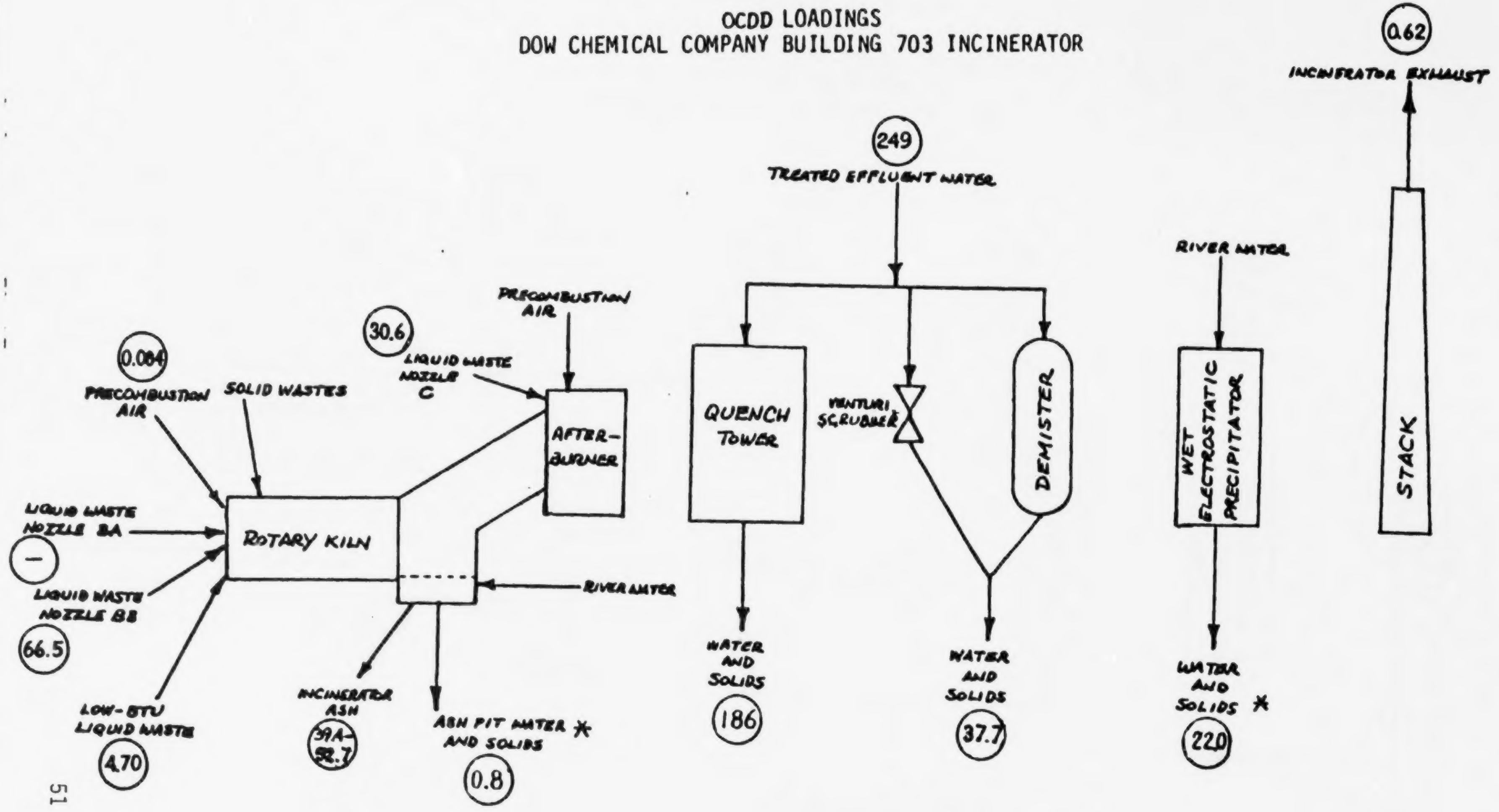
* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF TCDD

In		Out
218	(gm/yr)	87.3

FIGURE V-4

OCDD LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


0.62
INCINERATOR EXHAUST
249
TREATED EFFLUENT WATER
RIVER WATER
STACK
WET ELECTROSTATIC PRECIPITATOR
DEMISTER
VENTURI SCRUBBER
QUENCH TOWER
PRECOMBUSTION AIR
30.6
LIQUID WASTE NOZZLE C
AFTER-BURNER
0.084
PRECOMBUSTION AIR
SOLID WASTES
LIQUID WASTE NOZZLE 8A
—
ROTARY KILN
RIVER WATER
LIQUID WASTE NOZZLE 8B
66.5
LOW-BTU LIQUID WASTE
4.70
INCINERATOR ASH
39.4-52.7
ASH PIT WATER AND SOLIDS *
0.8
WATER AND SOLIDS
186
WATER AND SOLIDS
37.7
WATER AND SOLIDS *
220

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

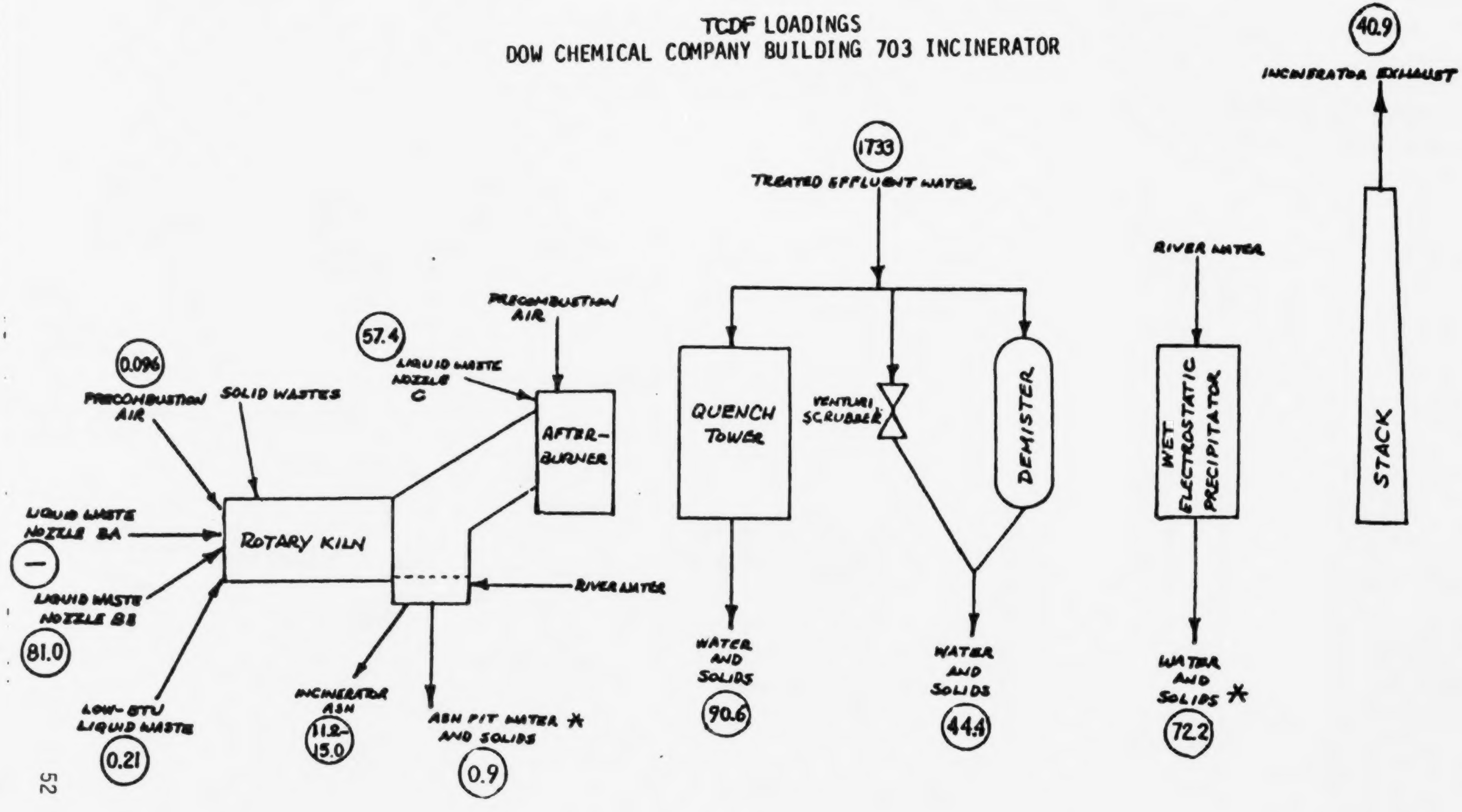
* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF OCDD

In		Out
351	(gm/yr)	286

FIGURE V-5

TCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


40.9
INCINERATOR EXHAUST
1733
TREATED EFFLUENT WATER
RIVER WATER
STACK
PRECOMBUSTION AIR
57.4
LIQUID WASTE NOZZLE C
0.096
PRECOMBUSTION AIR
SOLID WASTES
AFTER-BURNER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WET ELECTROSTATIC PRECIPITATOR
LIQUID WASTE NOZZLE 8A
—
ROTARY KILN
RIVER WATER
LIQUID WASTE NOZZLE 8B
81.0
WATER AND SOLIDS
90.6
WATER AND SOLIDS
44.4
WATER AND SOLIDS *
72.2
LOW-BTU LIQUID WASTE
0.21
INCINERATOR ASH
11.2-15.0
ASH PIT WATER AND SOLIDS *
0.9

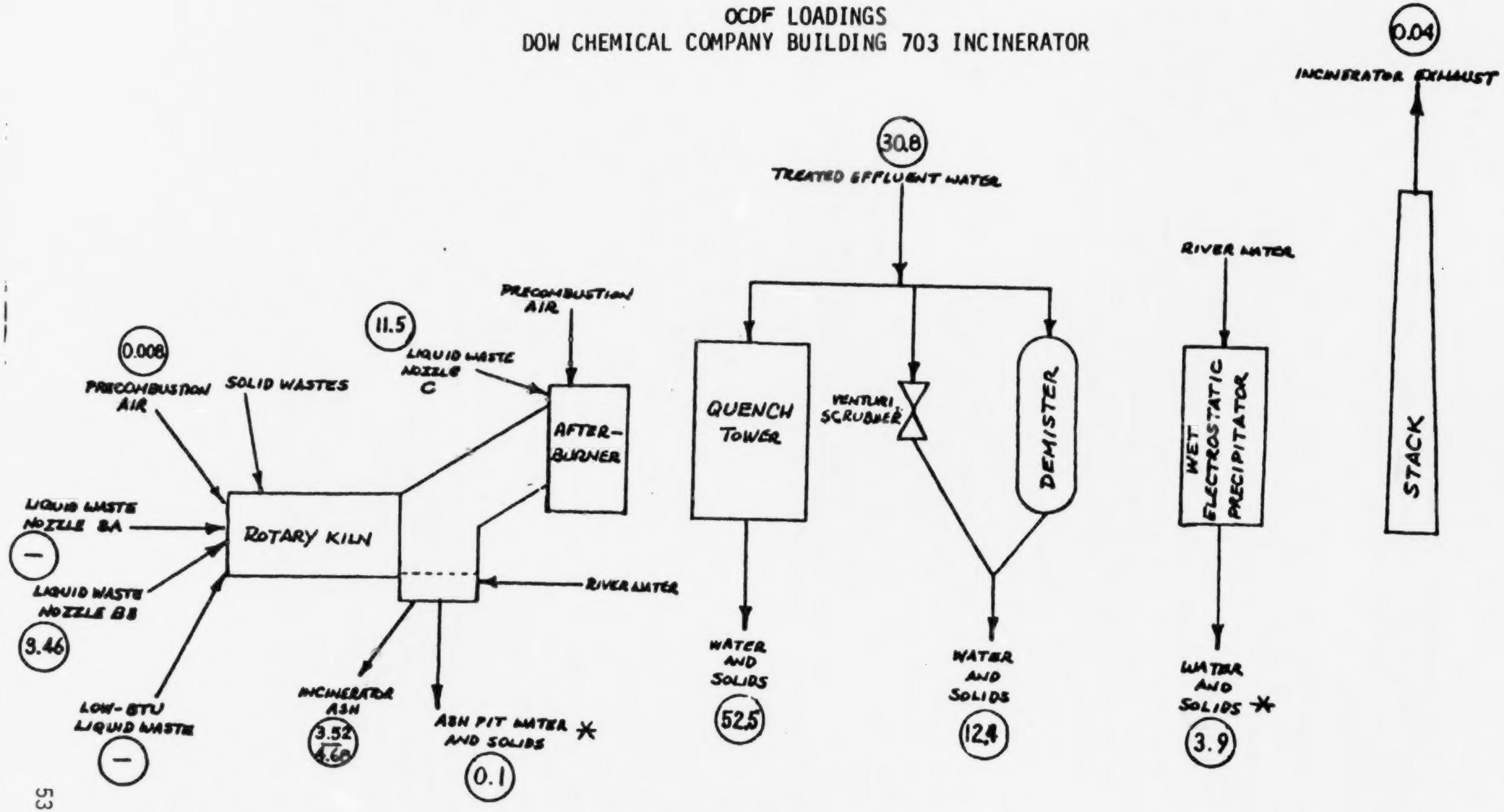
NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF TCDF		
In		Out
1872	(gm/yr)	238

FIGURE V-6

OCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


0.04
INCINERATOR EXHAUST
STACK
30.8
TREATED EFFLUENT WATER
RIVER WATER
WET ELECTROSTATIC PRECIPITATOR
PRECOMBUSTION AIR
11.5
LIQUID WASTE NOZZLE C
AFTER-BURNER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
0.008
PRECOMBUSTION AIR
SOLID WASTES
LIQUID WASTE NOZZLE 8A
—
ROTARY KILN
RIVER WATER
LIQUID WASTE NOZZLE 8B
3.46
LOW-BTU LIQUID WASTE
—
INCINERATOR ASH
3.52
4.60
ASH PIT WATER AND SOLIDS *
0.1
WATER AND SOLIDS
52.5
WATER AND SOLIDS
12.4
WATER AND SOLIDS *
3.9

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF OCDF

In		Out
45.6	(g/yr)	72

the ranges of these data frequently overlapped during the three sampling periods, and no consistent relationship appeared between any of these characteristics and the PCDD and PCDF concentrations appearing in exhaust air or discharged wastewaters or solids.

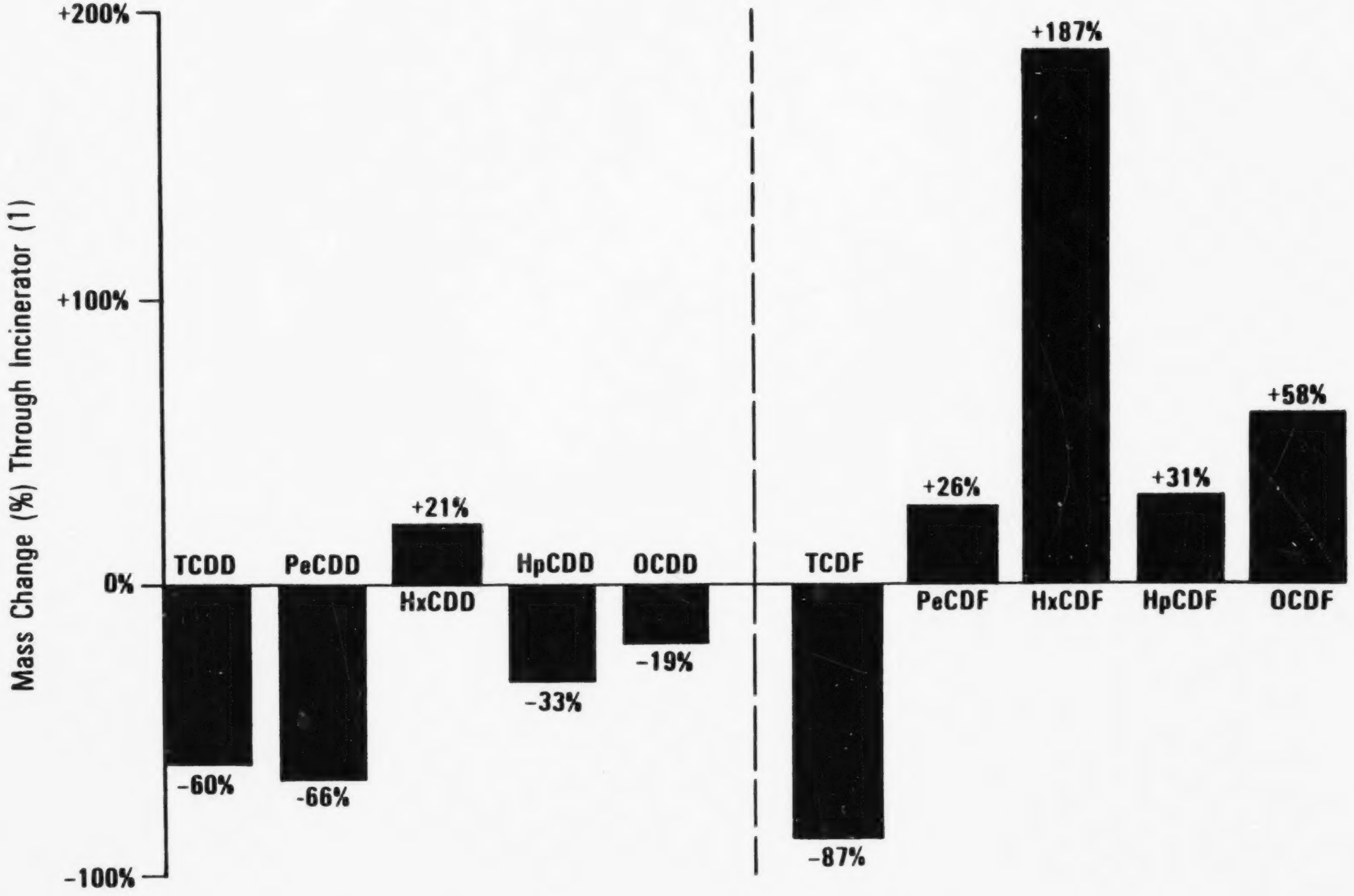
The waste feeds from nozzles "BB" and "C" varied widely over the three days, and there was no discernible characteristic in any waste which appeared to have direct bearing on the exhaust gas and wastewater PCDD and PCDF concentrations in Tables V-16 and V-20 through V-22. Referring to data appearing in Tables D-70 through D-72 in Appendix D, however, higher loadings of PCDDs and PCDFs in liquid wastes appear to translate into higher loadings in discharged streams. In particular, on the third day, loadings of discharged PCDDs and PCDFs were markedly lower, corresponding with lower loadings of PCDDs and PCDFs (and semi-volatile compounds and pesticides; see Tables V-8 and V-9) in liquid wastes. As indicated above, incinerator operational characteristics were similar on all three sampling days. For incinerator ash, there is no clear relation (see Table V-19), as considerably higher concentrations of all PCDD and PCDF homologues were found on the first day.

Figures V-3 to V-6 present a summary of annualized inputs and outputs of TCDD, OCDD, TCDF, and OCDF for the Building 703 incinerator. Figures D-1 through D-10 in Appendix D show these loadings for all PCDD and PCDF homologue groups. These estimates were calculated by averaging the mass inputs and outputs determined from the three test dates and converting the averages to annual discharges. Because not all input streams could be sampled (e.g., containerized waste, and loose refuse), the mass estimates are rough approximations. Nonetheless, the data may provide some interesting insights into the fate of PCDDs and PCDFs in the incinerator.

Figure V-7 compares the mass inputs and mass outputs. Negative values imply destruction of PCDDs and PCDFs; positive values imply formation in the incinerator. Values close to 0% change imply mass transfer from input streams to output streams. Those data suggest only limited destruction of TCDDs, somewhat higher destruction of PeCDDs (66%), and transfer of HxCDDs and OCDD from input streams to output streams. For PCDFs, the data suggest destruction of TCDFs (86%) and formation of HxCDFs and OCDF, and possibly PeCDFs and HpCDFs. However, a significant portion of some PCDD and PCDF homologue groups discharged from the incinerator appeared to have entered the incinerator system via the air pollution control device service water supplied from the Dow Chemical wastewater treatment facility. These PCDDs and PCDFs would not likely have been destroyed or altered in the once-through water systems serving the quench tower, venturi-demister, and ESP, or transferred to the incinerator exhaust gas stream. Within the bounds of this study, the extent to which PCDDs and PCDFs present in service water could have been destroyed, transferred to other streams, or increased with their passage through the incinerator system could not be evaluated. However, it is acknowledged that a portion of the PCDDs and PCDFs entering the incinerator air pollution devices may have returned largely unaltered to the Dow Chemical wastewater treatment system.

FIGURE V-7

DOW Chemical Company - Midland Plant
Building 703 Incinerator
Comparison of PCDD and PCDF Inputs and Outputs

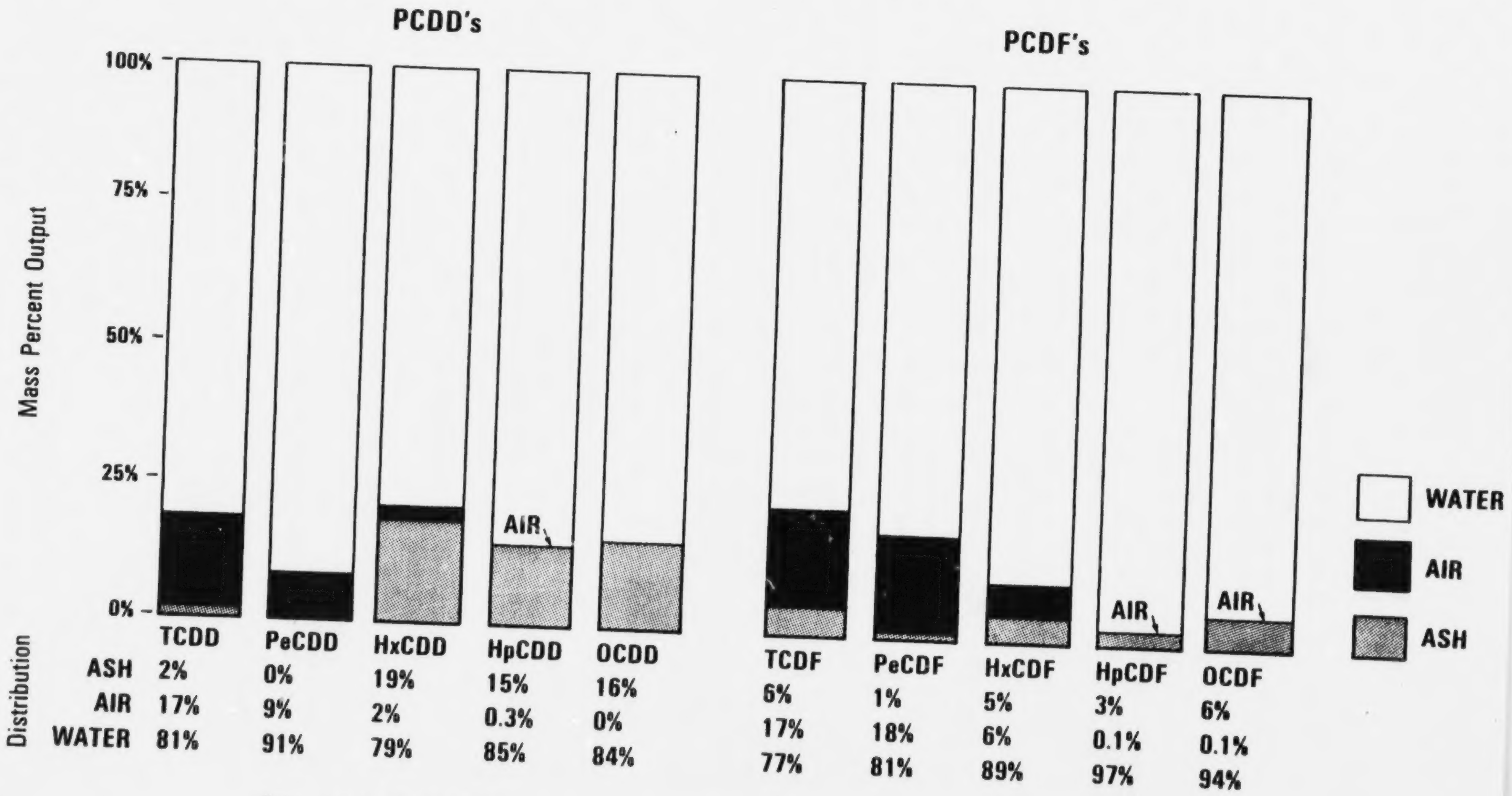


Note: (1) Defined as percent change from estimated mass inputs to estimated mass outputs.
Negative values imply destruction of PCDDs and PCDFs; positive values imply formation.
Values near zero imply mass transfer of PCDDs and PCDFs from input streams to output streams.

Figure V-8 shows the relative distribution of PCDD and PCDF homologues in air, water, and ash outputs from the incinerator. In all cases, most PCDDs and PCDFs were discharged in wastewaters; as indicated above, a significant loading of many PCDD and PCDF homologue groups entered the incinerator system via inlet water supplied to the air pollution control devices. Previous data tables appearing in this report show most of the PCDDs and PCDFs in wastewaters were found in filterable solids. For each homologue group, if the loadings found in wastewaters (primarily residing in filterable solids) are combined with those in discharged ash, it may be concluded that most, in terms of mass, are discharged along with solid effluents. Lower chlorinated homologues tend to appear in greater proportion in incinerator air exhausts; however, in this study, no more than 18% of any homologue group appeared in incinerator exhaust on a total loading basis.

FIGURE V-8

DOW Chemical Company - Midland Plant
Building 703 Incinerator
Distribution of PCDD's and PCDF's Among Incinerator Ash, Air, And Water Outputs



Note: Ash=Incinerator Ash Output
Air=Incinerator Exhaust Output
Water=Ash Pit, Quench Tower, Venturi Scrubber, Demister and Electrostatic Precipitator Water Outputs

VI. AMBIENT AIR SAMPLING STUDY IN VICINITY OF DOW CHEMICAL COMPANY MIDLAND PLANT

The Dioxin Strategy referenced in Section I of this report focused on seven types, or tiers, of locations and sources, ordered by decreasing potential for 2378-TCDD contamination. Combustion sources were grouped into Tier 4, for which sampling and analysis plans were formulated by EPA and published in February 1985 in a comprehensive project plan.^{4} That plan called for limited ambient air monitoring, only of precombustion air drawn into the combustion source. The ambient air sampling study in the vicinity of the Dow Chemical Company Midland Plant encompassed four sites at which monitors were operated to collect specific target compounds; the scope of the study thus went beyond that specified in the Tier 4 project plan. This was the only study conducted under Tier 4 program guidance at which extensive ambient air monitoring was done.

The sites were constructed and operated by a contractor, GCA/Technology Division, and arranged such that at least one of three sites would frequently be downwind of the Dow Chemical facility under typical summer wind conditions in the study area. Two of the downwind monitoring sites were selected as close as possible to the fenceline of the Dow Chemical plant. The third downwind site was placed in a residential and recreation area to assess compound concentrations to which the local population may be exposed. The fourth site was selected to be upwind of Dow Chemical under these conditions and would thus indicate background concentrations of the above compounds. Wind data were obtained at two sites near the monitoring network. Additional weather data were taken as needed from facilities maintained locally by Dow Chemical and from public sources operated by the National Oceanic and Atmospheric Administration (NOAA).

Monitoring was conducted between September 7 and 27, 1984, and included 18 days of sampling. Analyses of various types of samples were keyed to wind directions under which appropriate upwind-downwind relationships were experienced between monitoring stations. The site descriptions below include distances and directions with respect to the Building 703 liquid/solid waste incinerator as well as references to the Dow plant fenceline as it existed at the time of the study. While the primary focus of this study was the incinerator, which was in operation throughout the study period, the results are also indicative of numerous point source and fugitive emissions from the Dow Chemical plant.

The sampling network was designed to assess air quality impacts of the Dow Chemical plant, and was not intended to evaluate or determine the exact location of maximum effect. Also, the purpose of the network was to monitor the effects of the entire Midland Plant, rather than the Building 703 incinerator plume in particular. The frequency of plume impaction or fumigation at the monitoring sites was not evaluated, and the possible effects of phenomena such as downwash were not considered. However, two downwind monitoring sites were placed near the plant fenceline, where dispersion or dilution of plant emissions was likely to be lowest.

No dispersion modeling work was done prior to establishing the network, and the monitoring sites were, to a large extent, selected based upon the practicality of locating them on existing structures where physical obstructions to air flow were absent and adequate deliverable electrical power was available. These limitations, as well as the short duration of the ambient air study, should be borne in mind as the study results are evaluated.

Several months after the ambient air study was completed, ground-level exposure to PCDDs and PCDFs emitted from the stack of the Building 703 incinerator was estimated using the Human Exposure Model developed by USEPA. This model employed meteorological and population distribution data to determine the location of maximum impact to the surrounding population of a single point source. This analysis revealed the point of maximum plume impact to be 1 km northeast to east-northeast, downwind of the facility, close to sites 2 and 4 described below. A full discussion of this analysis, authored by David H. Cleverly of the Pollutant Assessment Branch, Strategies and Air Standards Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, appears in Appendix J.

A. Monitoring Network Description

Figure VI-1 shows the locations of each of the monitoring stations described below.

1. Site 1

As historical data from the Midland area indicated predominant summer winds to be from the south and southwest, this monitoring site was intended to be the upwind reference for the three stations located generally downwind the Dow facility. A two-meter-high equipment scaffold was placed on a low hill at the west end of Dow property, overlooking a series of Dow brine and wastewater treatment lagoons. The intersection of Ashby and Poseyville Roads was approximately 100 meters to the southwest; the Dow Chemical incinerator was located about 1.1 miles from the site at a heading of about 80°. Looking from the site, the Dow facility was visible in a sector extending between 0° and 105°; thus, winds blowing from any direction between 110° and 360° were considered not to have contacted any portion of the Dow Midland Plant prior to being sampled.

Site 1 included monitoring equipment for the following distinct groups of compounds:

PCDDs and PCDFs
Chlorobenzenes (principally Cl_2 through Cl_6)
Semi-volatile and volatile compounds (VOC)
Formaldehyde

Detailed descriptions of each of the above samplers appear later in this report. In addition to the above, site 1 was equipped with a wind speed and direction monitor; the sensors were placed at a height of 10 meters above ground. Figure VI-2 includes a site sketch and information concerning the inlet heights of the four samplers shown above.

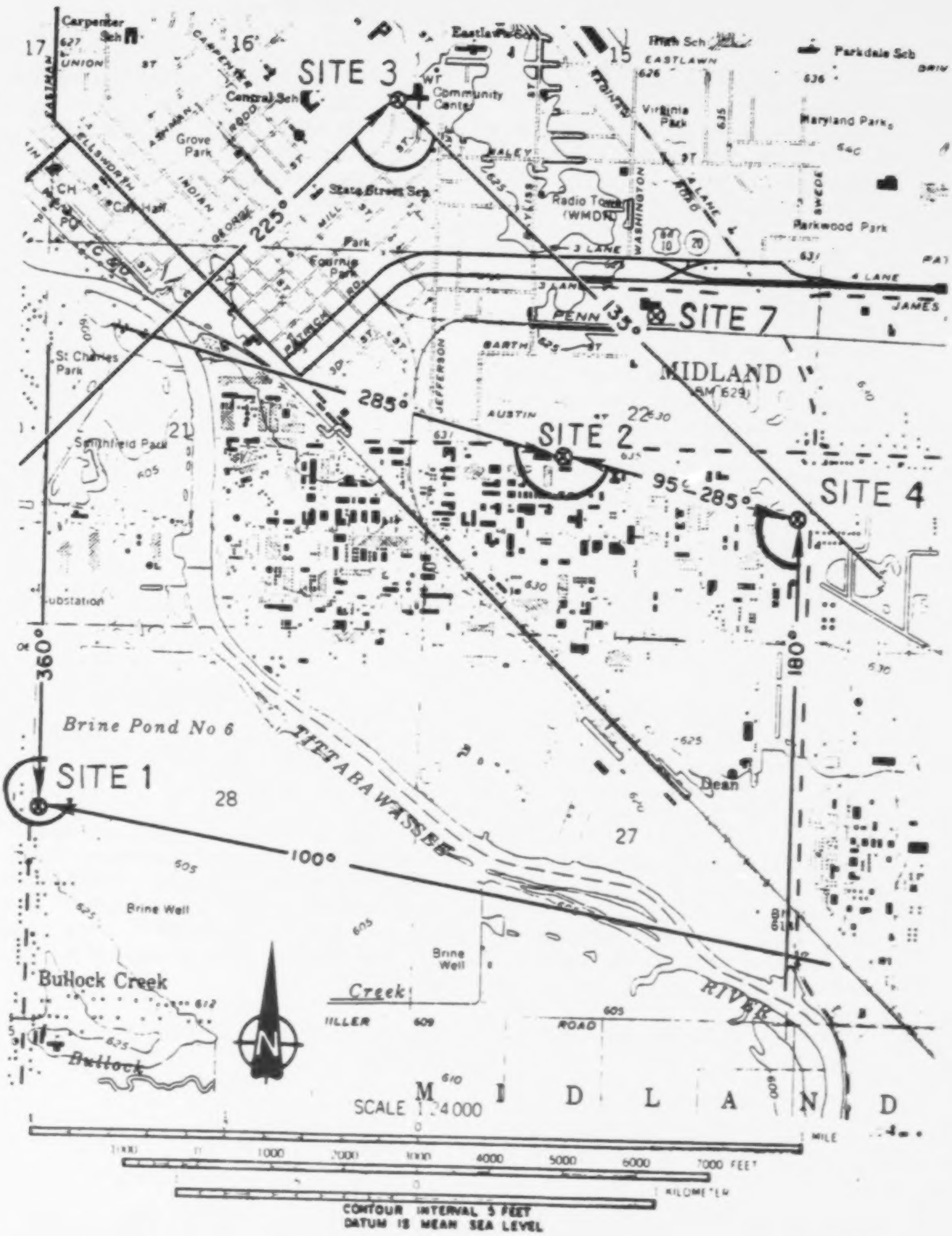
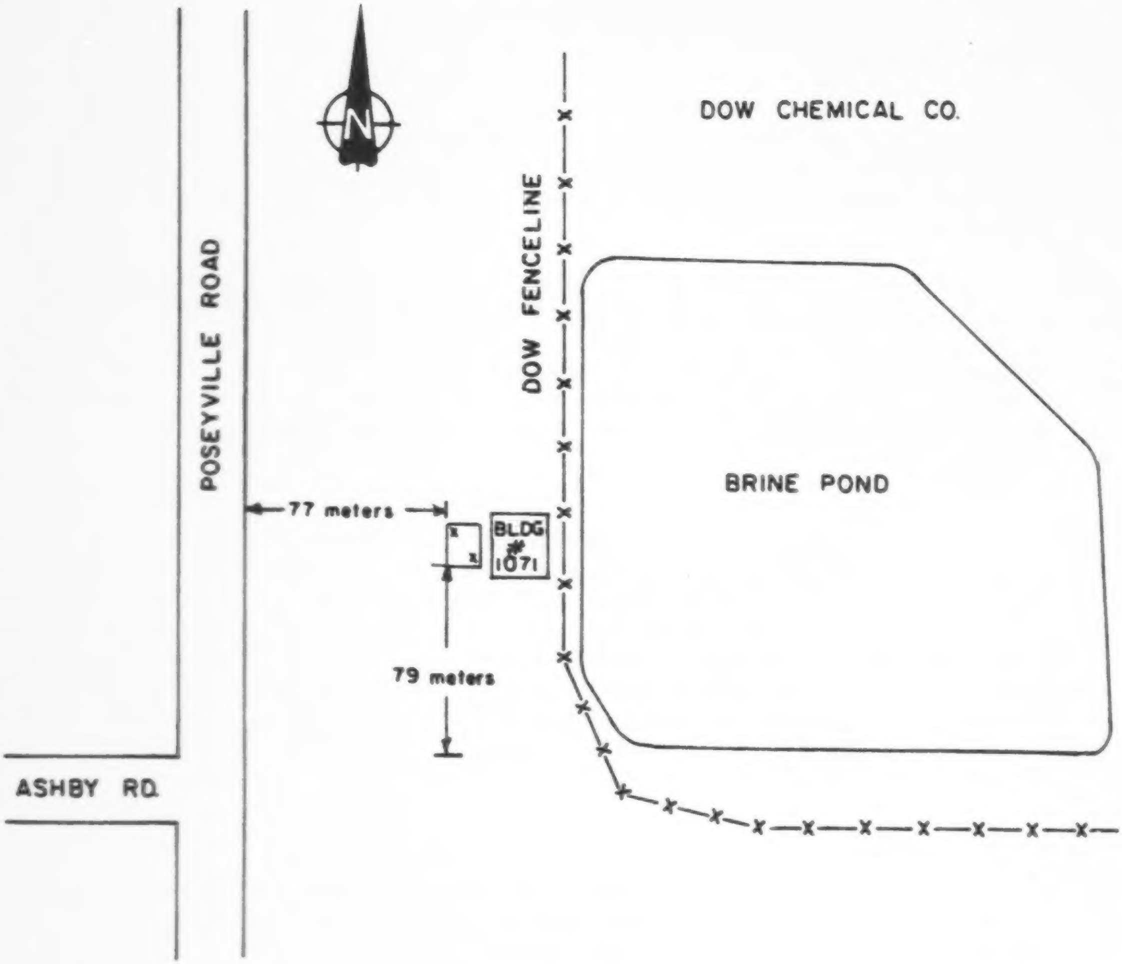

SITE 3
Community Center
SITE 7
SITE 2
SITE 4
SITE 1
225°
135°
285°
95°
285°
360°
180°
100°
MIDLAND
BM 629
Carpenter Sch
Central Sch
Grove Park
State Street Sch
Eastlawn Sch
High Sch
Parkdale Sch
Virginia Park
Maryland Park
Parkwood Park
Radio Tower (WMDN)
Fournie Park
St Charles Park
Smithfield Park
Substation
Brine Pond No 6
TITTABAWASSEE
RIVER
Brine Well
Brine Well
Bullock Creek
Bullock
Creek
Dean
MILLER
ROAD
EASTLAWN
UNION
ELLSWORTH
PENN
BARTH
AUSTIN
JEFFERSON
WASHINGTON
JAMES
3 LANE
4 LANE
17
16
15
21
22
28
27
M I D L A N D
SCALE 1:24000
MILE
1000 0 1000 2000 3000 4000 5000 6000 7000 FEET
KILOMETER
CONTOUR INTERVAL 5 FEET
DATUM IS MEAN SEA LEVEL

Figure VI-1

Midland, Michigan, Ambient Air Monitoring Network


DOW CHEMICAL CO.
N
POSEYVILLE ROAD
DOW FENCELINE
BRINE POND
77 meters
BLDG # 1071
79 meters
ASHBY RD.

1. Location - atop scaffolding adjacent to Dow Building No. 1071.
2. Nearest intersection - Poseyville and Ashby Roads, 110 meters to SW.
3. Pollutants monitored at this site - PCDD/PCDF, chlorobenzenes, VOCs, and formaldehyde.
4. Additional parameters monitored at this site - wind direction and wind speed.
5. Hi-Vol inlet height - 3.1 meters (PCDD/PCDF, chlorobenzenes).
6. CMS tube inlet height - 3.4 meters (VOCs).
7. Impinger inlet height - 2.6 meters (formaldehyde).
8. Meteorological equipment height - 10 meters (wind speed, wind direction).
9. Obstructions to samplers - none.
10. Orientation to Dow Chemical facility - Dow occupies the sector NE of the site, 0° N to 100° SE.
11. UTM coordinates - Zone 16; 4,829.9 km N; 722.1 km E.
12. Latitude/longitude - 43°35'25" N, 84°14'48" W.

Figure VI-2

Location of Ambient Air Monitoring Site 1

2. Site 2

This site was near the northern fenceline of the Dow facility, such that winds between 95° and 285° would pass through the plant before reaching it. The incinerator was about 0.8 mile from the site, at a bearing of 195°. A major east-west road, Bay City Road, passed about 16.5 meters to the north of the site.

Equipment was placed on the flat rooftop of Dow Building 911, a structure approximately 3.5 meters in height, to monitor the full range of compound groups as described for site 1. Sampler inlet heights are shown in Figure VI-3. In general, there were no significant obstructions to free air flow to the site; Dow Building 566, located about 45 meters southwest, was judged to be sufficiently distant to preclude significant wind eddying effects.

3. Site 3

To assess concentrations of target compounds in a population center and recreation area, site 3 was assembled atop the Midland Community Center, a flat-roofed multistory building about 0.9 mile north of the Dow Chemical fenceline and 1.8 miles from the plant incinerator. The incinerator was at a heading of 170° from site 3; however, winds between 135° and 225° were considered upwind with respect to the entire Dow facility. The site was configured as shown in Figure VI-4. An airflow obstruction cited in the figure was judged to be minor; in any event, this low wall was northwest of the monitoring equipment, not in the direction of emissions from the Dow facility.

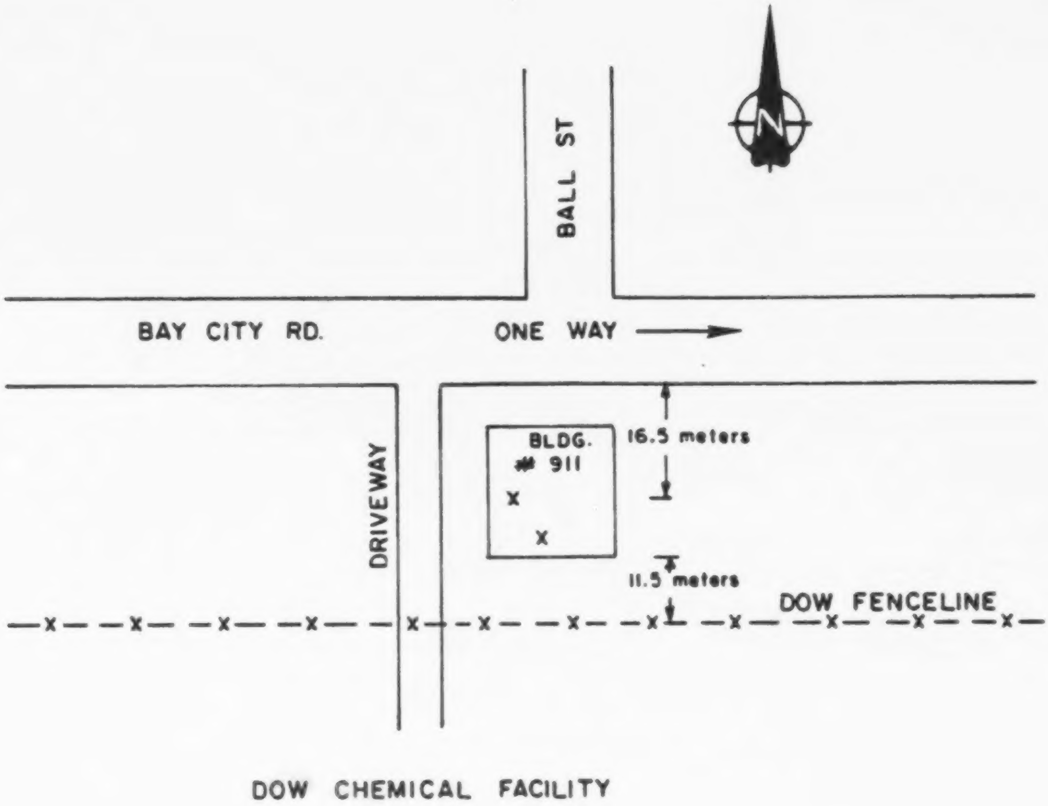
4. Site 4

A second site at the fenceline of the Dow plant was established to receive impacts from the facility under ambient wind conditions between 180° and 285°. The Building 703 incinerator was located 1.1 miles from the monitoring station, at a heading of 230°. Monitoring equipment was placed atop a mobile laboratory trailer parked in a lot located at the east boundary of the Dow Chemical facility. Sampler inlet heights are shown in Figure VI-5. The site was selected to deploy field duplicate and field blank samples because of the ease of servicing this site with equipment stored in the trailer. Periodic weather data (temperature, relative humidity, and barometric pressure) were obtained manually at this site.

5. Other Sites

A fifth site, designated as site 7 (Figure VI-6), consisted of a monitoring trailer operated continuously by the Michigan Department of Natural Resources, and included wind speed and direction measurement equipment. The Dow Chemical north fenceline was about 0.3 mile south of this location. Wind data gathered at the site were considered equivalent to those at sites 2 and 4 and were used as a check on similar data at site 1.

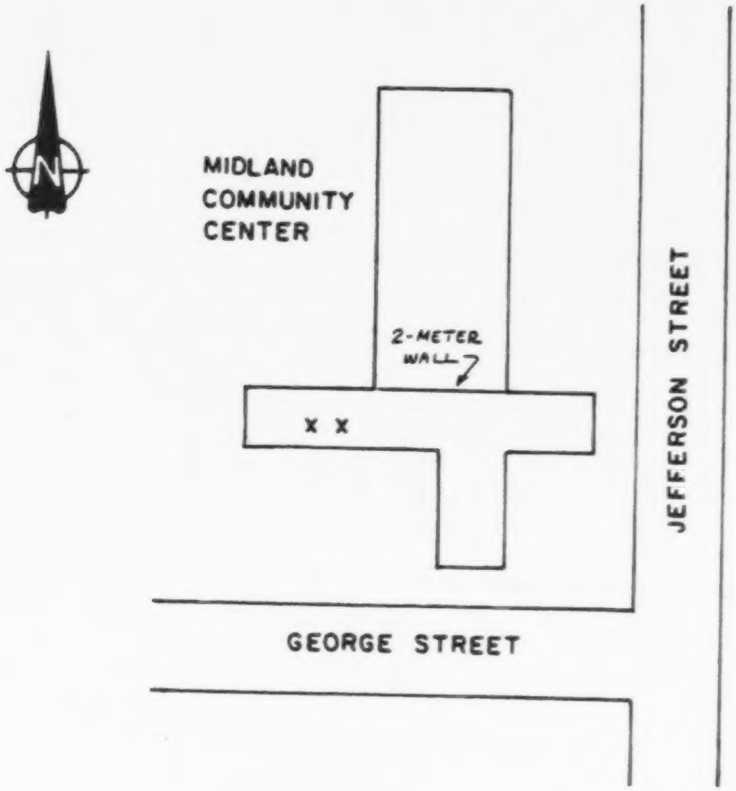
Sites 5 and 6 were planned as meteorological stations, but were not used and are not shown in Figure VI-1.


BALL ST
BAY CITY RD.
ONE WAY
DRIVEWAY
BLDG. # 911
16.5 meters
11.5 meters
DOW FENCELINE
DOW CHEMICAL FACILITY

1. Location - atop Dow Building No. 911.
2. Nearest intersection - Bay City Road and Ball Street, adjacent to site.
3. Pollutants monitored at this site - PCDD/PCDF, chlorobenzenes, VOCs, and formaldehyde.
4. Additional parameters monitored at this site - none.
5. Hi-Vol inlet height - 4.9 meters (PCDD/PCDF, chlorobenzenes).
6. CMS tube inlet height - 4.9 meters (VOCs).
7. Impinger inlet height - 4.1 meters (formaldehyde).
8. Obstructions to samplers - possible obstruction is building approximately 45 meters SW of sample.
9. Orientation to Dow Chemical facility - Dow occupies the sector from 95° SE to 285° NW.
10. UTM coordinates - Zone 16; 4,831.4 km N; 724.2 km E.
11. Latitude/longitude - 43°36'17" N, 84°13'14" W.

Figure VI-3

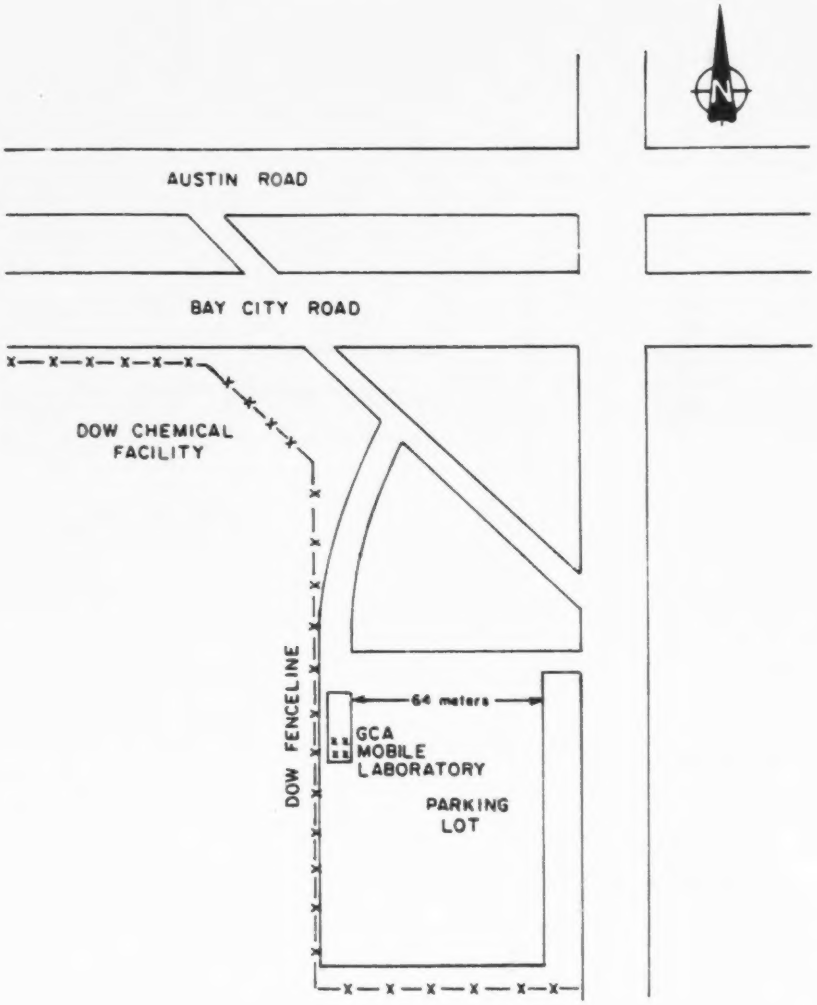
Location of Ambient Air Monitoring Site 2



1. Location - atop roof of Midland Community Center.
2. Nearest intersection - George St. and Jefferson St., adjacent to building.
3. Pollutants monitored at this site - PCDD/PCDF, chlorobenzenes, VOCs, and formaldehyde.
4. Additional parameters monitored at this site - none.
5. Hi-Vol inlet height - 5.7 meters (PCDD/PCDF, chlorobenzenes).
6. CMS tube inlet height - 6.0 meters (VOCs).
7. Impinger inlet height - 5.2 meters (formaldehyde).
8. Obstructions to samplers - possible obstruction is a 2 meter brick wall approximately 10 meters to the NW.
9. Orientation to Dow Chemical facility - Dow occupies the sector 135° SE to 225° SW.
10. UTM coordinates - Zone 16; 4,832.9 km N; 723.6 km E.
11. Latitude/longitude - 43°37'04" N, 84°13'41" W.

Figure VI-4

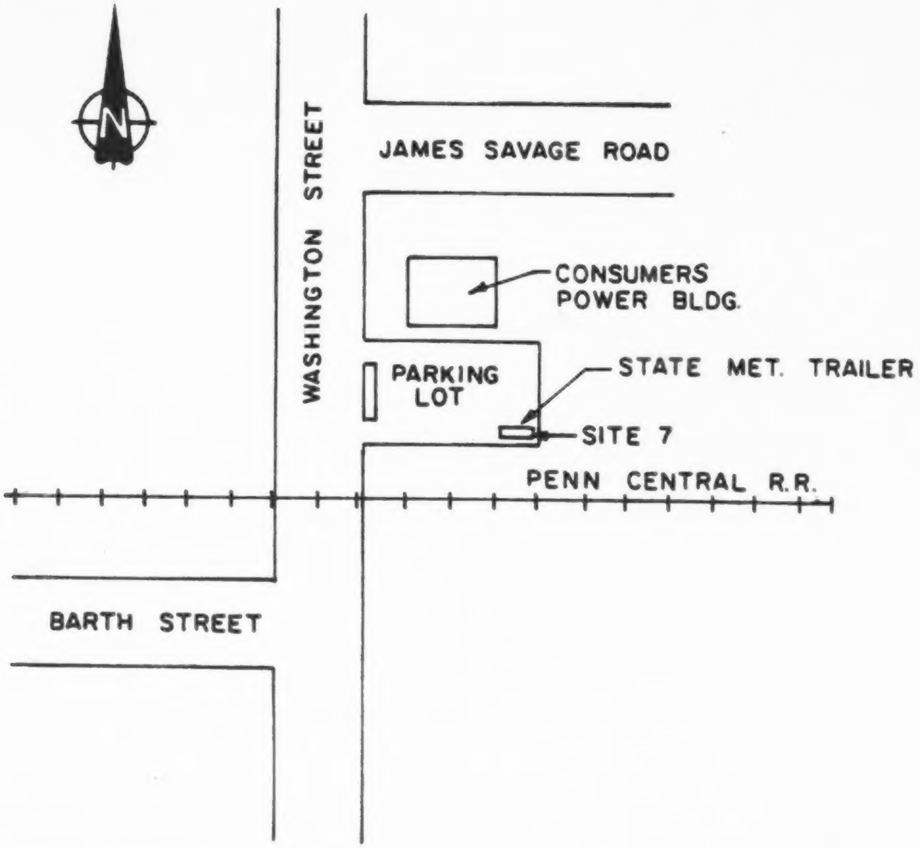
Location of Ambient Air Monitoring Site 3


AUSTIN ROAD
BAY CITY ROAD
DOW CHEMICAL FACILITY
DOW FENCELINE
64 meters
GCA MOBILE LABORATORY
PARKING LOT
N

1. Location - located atop GCA Mobile Laboratory in NE parking lot.
2. Nearest intersection - Bay City Road and S. Saginaw Road.
3. Pollutants monitored at this site - PCDD/PCDF, chlorobenzenes, VOCs, and formaldehyde.
4. Additional parameters monitored at this site - temperature, barometric pressure, and relative humidity.
5. Hi-Vol inlet height - 5.1 meters (PCDD/PCDF and chlorobenzenes).
6. CMS tube inlet height - 5.4 meters (VOCs).
7. Impinger inlet height - 4.6 meters (formaldehyde).
8. Obstructions to samplers - none.
9. Orientation to Dow Chemical facility - Dow occupies the sector 180° S to 285° NW.
10. UTM coordinates - Zone 16; 4,831.2 km N; 725.2 km E.
11. Latitude/longitude - 43°36'09" N, 84°12'28" W.

Figure VI-5

Location of Ambient Air Monitoring Site 4


N
WASHINGTON STREET
JAMES SAVAGE ROAD
CONSUMERS POWER BLDG.
PARKING LOT
STATE MET. TRAILER
SITE 7
PENN CENTRAL R.R.
BARTH STREET

1. Location - Michigan DNR trailer in parking lot of Consumers Power on Washington Street.
2. Nearest intersection - James Savage Road and Washington Street.
3. Parameters at this site - wind direction and wind speed.
4. UTM coordinates - Zone 16; 4,832.0 km N; 724.6 km E.

Figure VI-6

Location of Ambient Air Monitoring Site 7 (Wind Monitoring Site)

B. Monitor Descriptions and Sampling Methods

All four of the monitoring sites included equipment to monitor four groups of compounds: PCDDs and PCDFs; higher-substituted chlorobenzenes (Cl_2 through Cl_6); a general range of semi-volatile and volatile compounds; and formaldehyde. The samplers specific to each group are described in detail in Appendix E to this report.

C. Conduct of Study

1. Sampling Procedures

Field methods for the four types of 24-hour samplers employed in this study (modified high-volume sampler for PCDDs and PCDFs, and chlorobenzenes and other semi-volatile compounds; carbon molecular sieve sampler for volatile compounds; and impinger-type sampler for formaldehyde) were taken from the literature and modified as necessary according to meteorological conditions encountered, and the limitations of the selected analytical laboratories. While it would have been preferable to operate all four sampler types at each site on every day, practical and resource limitations led to decisions under which some samplers were run only during periods when meteorology was favorable (good upwind-downwind relationships existed), or a limited number of exposed samples were designated for analysis. These decisions are described in the detailed discussion of sampling methods appearing in Appendix E, and a summary of samples obtained is presented in Table VI-1. Preparation and assembly of sampler materials were for the most part coordinated in the GCA sampling trailer also used as monitoring site 4.

Detailed descriptions of sampling procedures for all of the ambient air monitors used in this study may be found in Appendix E of this report.

2. Custody, Sample Handling, and Shipping

Samples were obtained and identified using chain-of-custody procedures described in the Quality Assurance Project Plan developed for the study,[15] and EPA custody forms and GCA data record forms shown in Appendix D of Reference 16 of this report. In short, standard EPA chain-of-custody protocols were followed in the conduct of work.

Cleaned and prepared sampling media, with the exception of DNPH reagent for formaldehyde sampling, were held in a secured trailer (site 4) until use. As indicated in Appendix E, DNPH reagent was prepared immediately before use and shipped to the study area for placement in sampling equipment. Exposed sampling media were kept in secured (locked or sealed) chests, separated from unexposed media, in the site 4 monitoring trailer before shipping. Subject to appropriate holding times, samples were shipped under EPA custody procedures and documents specific to the EPA Special Analytical Services program, to the contract laboratories selected to perform analyses for various compound classes. For volatile and semi-volatile compounds, and formaldehyde, analytical services were provided by United States Testing Company, Hoboken, New Jersey. For PCDD and PCDF, analyses were conducted by Midwest Research Institute, Kansas City, Missouri.

TABLE VI-1

MIDLAND, MICHIGAN AMBIENT AIR SAMPLING STUDY
SUMMARY OF SAMPLE TYPES AND SAMPLING TIMES

Run Start Date	PCDD/PCDF	Chlorobenzenes Semi-Volatiles	Volatiles	Formaldehyde
9/7/84		X	X	X
9/8	X	X	X	X
9/9				
9/10				
9/11		X		
9/12	X	X	X	X
9/13		X		
9/14		X		
9/15		X		
9/16		X		
9/17		X	X	
9/18		X	X	X
9/19		X	X	X
9/20		X		
9/21		X		
9/22	X	X	X	
9/23		X	X	
9/24		X	X	
9/25		X		
9/26		X		

NOTE: X denotes sample taken and submitted for analysis.

D. Analytical Procedures and Quality Assurance

Analytical methods specified for this study appear in References 17 (PCDD/PCDF) and 18 (semi-volatile compounds, volatile compounds, and formaldehyde), and are summarized briefly below:

PCDD/PCDF and Semi-Volatile Compounds	- Extraction followed by solvent partitioning and liquid chromatography, analysis by gas chromatography/mass spectrometry.
Volatile Compounds	- Collection on carbon molecular sieves, then thermal desorption and analysis by GC/MS.
Formaldehyde	- Reverse phase high performance liquid chromatography.

Samples collected during this study were identified, packed (cooled as appropriate), and shipped via commercial services for next-day arrival at contract laboratories. Selection of contract laboratories referenced in Section VI.C was coordinated by the USEPA Region V Central Regional Laboratory. Analytical data returned from the contract laboratories were reviewed for consistency with contract requirements by the USEPA Sample Management Office (Viar and Company, Alexandria, Virginia), and for adherence to quality assurance criteria contained in the Quality Assurance Project Plan for this study (see Reference 15) by the USEPA Region V Central Regional Laboratory. The results of these reviews are referenced in the discussion of general analytical findings which follows as Section VI.E of this report.

E. Results of Study and Discussion

1. PCDD/PCDF

Consistent with the evaluation of incinerator exhausts, a range of recovery of analytical surrogate or internal standard compounds of 50% to 150% was considered acceptable with respect to the suitability of PCDD and PCDF data. Recoveries of internal standards for PCDDs and PCDFs ranged between 22% and 220%, with no reportable recovery in a small number of cases.

Four internal standards were used: $^{13}C_{12}$ 2378-TCDD, $^{13}C_{12}$ 2378-TCDF, $^{37}Cl_4$ 1,2,3,4,6,7,8-HpCDD, and $^{13}C_{12}$ OCDD. Overall performance with respect to recoveries within the acceptable range of 50% to 150% was as follows for the 45 samples included in these analyses:

	Percent of Samples Within Acceptable Range
$^{13}C_{12}$-2378-TCDD	82% (37/45)
$^{13}C_{12}$-2378-TCDF	89% (40/45)
$^{37}Cl_4$-1,2,3,4,6,7,8-HpCDD	71% (32/45)
$^{13}C_{12}$-OCDD	80% (36/45).

The standard $^{13}C_{12}$ 2378-TCDD is of primary importance as the accuracy determinant for tetra- through hexa-CDD; those homologue groups are of greatest priority in assessing potential risks to health. In the above table, satisfactory recoveries were experienced in 82% of the samples.

Recoveries of the other three standards serve to measure analytical accuracies for PCDD and PCDF homologues which are of lesser concern with respect to health risk assessment. In summary, considering the low levels of detection specified for this study (parts per quadrillion in air), the data presented below are reasonably complete in terms of accuracy.

Complete results of sampling for PCDD and PCDF for the three selected sampling days are presented in Table VI-2; these were derived from the raw data shown in Appendix G, Table G-1, which are as received from the analytical laboratory. Two of the glass filter (polyurethane foam plug sample pairs (from sites 2 and 3 on September 8 and 9, 1984) analyzed by Midwest Research Institute (MRI) were reanalyzed for verification by the Environmental Monitoring and Support Laboratory (EMSL) of EPA in Research Triangle Park, North Carolina. The following findings were stated in the EMSL reanalysis and review report:

- Standards values were in reasonable agreement,
- Quantification of PCDD and PCDF appeared generally accurate,
- Most of the TCDF detected in the samples were 1238, 1467, 2468, and 1236 isomers, which were indicated by the EMSL as having been detected previously in incineration process samples from other studies,
- Similar isomer groups were found in samples of soils which were analyzed as part of a previous EPA Region V sampling program conducted in Midland, Michigan, in 1984,
- Between 20 and 50% of the concentration of PeCDFs reported in the samples was attributable to chlorinated diphenylethers (CDEs) which elute simultaneously from the capillary column used in analysis (co-elution of other CDEs with other PCDFs was not investigated, and
- The analytical results should be considered minimum values as the air sampling method employed in this study was not formally validated as of the time the study occurred.

Note in Table VI-2 that 2378-TCDD and 2378-TCDF were not detected by MRI in any sample. In the EMSL reanalyses, however, both isomers were found, as shown in the raw data in Appendix H. In two of three cases in which the EMSL reported values where MRI did not, the levels of 2378-TCDD and 2378-TCDF detected by the EMSL were above the detection limits stated by MRI. These data are presented in Table VI-3. The single finding of 2378-TCDD, in the sample from site 2 on 9/8-9/84, would result in an ambient air concentration of about 4.8 pg/m^3 (ppq).

In Table VI-4, the comparative results of analyses for TCDD and TCDF by MRI and the EMSL are presented in terms of concentration in air. These data show generally close agreement. The full text of the EMSL's description of reanalysis of these samples is presented in Appendix H.

TABLE VI-2

RESULTS OF AMBIENT AIR PCDD/PCDF SAMPLING IN VICINITY OF DOW CHEMICAL COMPANY, MIDLAND, MICHIGAN, SEPTEMBER 1984

(All data stated in picograms per cubic meter.)

Sample Identification		2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
9/8-9/84													
Average wind	Site 1	ND^1	0.99	ND^1	0.95	0.81	1.15	ND^1	0.86	ND^1	ND^1	ND^1	ND
199°, 6 mph	Site 2	ND(0.85)	44.80	9.28	ND(0.84)	2.08	7.70	ND(0.84)	249.80	29.80	4.15	5.01	3.42
	Site 3	ND(0.22)	2.40	ND(0.46)	ND(0.32)	2.07	7.92	ND(0.34)	14.72	4.44	ND(0.37)	ND(0.79)	1.36
	Site 4*	ND(0.09)	0.86	ND(0.09)	0.86	1.00	2.69	ND(0.12)	1.53	1.16	ND(0.65)	ND(0.52)	1.66
	Site 4 duplicate	ND(0.15)	0.48	ND(0.31)	ND(1.11)	1.54	4.10	ND(0.17)	2.70	1.41	0.73	ND(1.15)	0.84
	Precision (RPD)	--	56.7	--	--	42.5	41.5	--	55.3	19.5	--	--	65.6
9/12-13/84													
Average wind	Site 1	ND(0.19)	0.13	ND(0.38)	ND(1.02)	0.69	1.66	ND(0.18)	14.52	ND(2.93)	ND(0.62)	ND(2.16)	0.99
191°, 6 mph	Site 2	ND(0.24)	ND^2	ND(0.43)	ND(2.55)	ND(3.51)	ND(6.71)	ND(0.24)	14.53	ND(1.07)	ND(1.02)	ND(1.92)	ND(3.35)
	Site 3	ND(1.07)	3.27	ND(0.80)	ND(1.19)	0.65	5.10	ND(0.24)	44.95	2.22	ND(1.31)	ND(1.24)	0.81
	Site 4	ND(0.15)	0.38	ND(0.15)	2.93	1.48	6.75	ND(0.20)	13.88	1.06	ND(1.27)	ND(0.90)	2.67
	Site 4 duplicate	ND(0.17)	ND^2	ND(0.64)	ND(1.39)	0.48	5.60	ND(0.17)	11.21	3.01	ND(0.80)	ND(5.43)	ND(3.40)
	Precision (RPD)	--	--	--	--	102.0	18.6	--	21.2	95.8	--	--	--
9/22-23/84													
Average wind	Site 1	ND(0.06)	ND^2	ND(0.24)	ND(0.18)	ND(0.69)	0.30	ND(0.11)	ND^2	ND(0.13)	ND(0.26)	ND(0.83)	0.13
212°, 5 mph	Site 2	ND(0.05)	22.35	ND(0.32)	0.55	2.69	14.29	ND(0.99)	155.69	7.45	4.52	2.93	1.60
	Site 3	ND(0.08)	0.59	ND(0.48)	ND(0.39)	0.55	2.73	ND(0.12)	2.14	ND(0.23)	ND(0.15)	ND(0.80)	0.70
	Site 4	ND(1.63)	74.07	1.37	0.28	1.14	4.01	ND(1.63)	375.37	36.73	3.00	3.00	4.64
	Site 4 duplicate*	*ND(0.59)	24.28	ND(1.17)	0.96	1.41	4.37	ND(1.41)	122.70	15.42	4.37	2.70	6.55
	Precision (RPD)	--	101.3	--	109.7	21.2	8.6	--	101.5	81.7	37.2	10.5	34.1

*Denotes analysis of polyurethane foam plug was not provided by analytical laboratory. Calculation of analytical precision should therefore be considered tentative.

"ND" symbol indicates isomer or homologue was not detected at method detection limit. The higher of the two detection limits (for glass fiber filter or PUF plug) is stated.

[1] Detection limit not determined.

[2] Exposed sample concentration lower than that in field blank. Consider equivalent to nondetectable.

TABLE VI-3

COMPARATIVE ANALYSES FOR TOTAL AND 2378 ISOMER OF TCDD AND TCDF MIDWEST RESEARCH INSTITUTE AND EMSL-RTP, EPA

Sample Identification		Amount Detected (ng/sample)	
		MRI	EMSL
9/8-9/84, Site 2 Filter	2378-TCDD	ND (0.10)	0.4
	Total TCDD	3.7	9.0
	2378-TCDF	ND (0.69)	0.2
	Total TCDF	36	28.0
9/8-9/84 Site 2 PUF	2378-TCDD	ND (0.70)	ND
	Total TCDD	33	29.0
	2378-TCDF	ND (0.40)	ND
	Total TCDF	180	131.0
9/8-9/84 Site 3 Filter	2378-TCDD	ND (0.18)	ND
	Total TCDD	1.6	0.8
	2378-TCDF	ND (0.20)	ND
	Total TCDF	7.5	2.2
9/8-9/84 Site 3 PUF	2378-TCDD	ND (0.12)	ND
	Total TCDD	1.7	1.4
	2378-TCDF	ND (0.28)	0.4
	Total TCDF	3.9	26.0

Note: ( ) Detection limit expressed in nanograms.

TABLE VI-4

COMPARATIVE VALUES FOR 2378-TCDD, TOTAL TCDDs, 2378-TCDF, and TOTAL TCDFs MIDWEST RESEARCH INSTITUTE AND EMSL-RTP, EPA

	9/8-9/84, Site 2				9/8-9/84, Site 3			
	2378-TCDD	Total TCDDs	2378-TCDF	Total TCDFs	2378-TCDD	Total TCDDs	2378-TCDF	Total TCDFs
EMSL-EPA								
Filter	0.49	11.00	0.24	34.21	ND	0.97	ND	2.68
PUF	ND	35.43	ND	160.06	ND	1.71	0.49	31.66
Total	0.49	46.43	0.24	194.27	ND	2.68	0.49	34.34
MRI*								
Total	ND	44.80	ND	249.80	ND	2.40	ND	14.72

*Taken from Table VI-2. Data stated in pg/m^3.

Along with the above reanalysis, the data provided by MRI were reviewed by the EPA Region V Central Regional Laboratory. Following are the principal findings of that review, as they relate to the quality of these data:

- The surrogate compound $^{37}Cl_4$-2378-TCDD was not added to any sample, as required by the analytical specifications for this study. With this lacking, MRI provided internal standard recovery data by quantitating one internal standard against another. The recoveries of the surrogate $^{13}C_{12}$-TCDF were considered as indication of bias for tetra- and penta-CDD and CDF; an overall bias of -13% was found.

  Based on recoveries of the surrogate $^{37}Cl_4$-HpCDD, the bias for hexa- through octa-CDD and CDF was calculated to be +11%. Both of these biases were considered small with respect to the errors introduced by taking the recovery of a particular homologue to represent that of a different homologue.

- Field blank samples were spiked to calculate recoveries and precision, and five of the 42 analyses showed spike recoveries out of control. However, precision criteria were met in the duplicate blanks.

  Since all field blank samples were spiked by MRI, it was not possible to estimate possible field contamination as planned in the analytical protocol. However, in the spiked blanks, the levels detected were close to the spiking levels, suggesting field contamination was not significant.

- While the analytical request called for a laboratory matrix spike for every ten samples analyzed, this was not provided. This was judged to be a minor shortfall, and available matrix spike data showed generally satisfactory performance.

- Resolution of 2378-TCDD from neighboring TCDDs ranged between 40 and 60%; the analytical request specified that samples were to have been rerun if resolution was 25% or greater. As MRI did not detect 2378-TCDD in any sample, but the EMSL did, this implies that some of that reported by MRI as total TCDDs may in fact have been 2378-TCDD.

- Response factors calculated by MRI for some calibration standards were not substantiated by verifiable data. Most were provided, however, and indicated satisfactory performance.

In summary, the Central Regional Laboratory review of the MRI data package indicated the data were generally suitable for project use, as qualified above.

Wind data for the duration of the ambient air sampling study are presented in Table VI-5. As indicated previously, three of the periods having most favorable upwind-downwind alignment of monitoring sites with respect to the Dow Chemical facility were chosen for PCDD and PCDF sample analyses. Wind conditions averaged over each of these three periods are stated in Table VI-2; Figure VI-1 may be used to relate these wind directions to the findings of PCDD and PCDF shown in Table VI-2.

From these data, it is apparent that site 1 was upwind of the Dow Chemical facility on all three days; correspondingly, the lowest concentrations of nearly all PCDD and PCDF homologues were detected at this site. Higher concentrations were consistently found at those sites downwind of the Dow facility. For the first two sampling periods analyzed, these were sites 2 and 3, while on the third sampling day, sites 2 and 4 were highest in most homologues.

On the first sampling day, highest concentrations were detected at the north fenceline of the Dow facility, with considerably less found at the comparatively distant Midland Community Center site. Under very similar wind conditions in the second sampling period, however, this pattern reversed, with concentrations of most PCDD and PCDF homologues in the same range (1 to 10 pg/m^3) on both days. With winds shifted 15 to 20 degrees toward the southwest on the third sampling day, highest concentrations were found exclusively at the two Dow Chemical fenceline sites. Precision between duplicate samples on all three days was frequently within the target range of ± 50% (relative percent difference).

Overall, these data establish that point and fugitive emissions of PCDD and PCDF from the Dow Chemical plant may be detected at downwind monitoring locations. Downwind concentrations were consistently higher than those upwind of Dow Chemical.

In Table VI-6, the concentration data in Table VI-2 are presented in terms of the portions of the PCDD and PCDF homologues found in the glass fiber filter and polyurethane foam plug of the samplers. These data suggest that the lower-chlorinated homologues, chiefly the tetra- through penta-, tend to reside in the polyurethane foam plug, while the hexa- through octa- homologues are principally found on the first-stage glass fiber filter, where more particulate matter is likely to be caught. These findings imply that

- higher-chlorinated homologues of PCDD and PCDF may bind selectively to particulate matter, while the tetra- and penta- homologues remain in the gaseous state or bound to finer particulates. These lower-chlorinated homologues may not be trapped efficiently by the glass fiber filter portion of the high-volume sampler, or may be air-stripped from the filter catch by the action of air moving through the sampler; and
- both components of the high-volume sampler should be used in series to determine the concentration of the full range of PCDD and PCDF homologues.

TABLE VI-5

WIND DATA - AMBIENT AIR SAMPLING PROGRAM
MIDLAND, MICHIGAN - SEPTEMBER 7-27, 1984

Run dates	GCA Run No.	EPA Run No.	Wind Direction		Wind Speed	
			Mean, degrees	Std. deviation	Mean, mph	Std. deviation
9/7-8	3	84ET08	184	12	5.9	1.5
9/8-9	4	84ET09	199	14	6.2	2.1
9/11-12	5	84ET10	329	91	3.8	0.9
9/12-13	6	84ET11	191	40	5.6	1.3
9/13-14	7	84ET12	309	32	3.8	1.3
9/14-15	8	84ET13	331	25	6.6	1.6
9/15-16	9	84ET14	296	62	4.9	2.8
9/16-17	10	84ET15	257	38	3.3	2.4
9/17-18	11	84ET16	212	9	4.1	1.5
9/18-19	12	84ET17	235	30	4.0	2.0
9/19-20	13	84ET18	250	44	4.1	1.5
9/20-21	14	84ET19	334	41	3.7	2.0
9/21-22	15	84ET20	12	134	4.1	1.7
9/22-23	16	84ET21	212	15	4.9	2.1
9/23-24	17	84ET22	197	42	2.6	1.1
9/24-25	18	84ET23	195	25	4.9	1.4
9/25-26	19	84ET24	284	25	6.1	1.9
9/26-27	20	84ET25	293	31	2.7	1.4

TABLE VI-6

RESULTS OF AMBIENT AIR SAMPLING FOR PCDD/PCDF
IN VICINITY OF DOW CHEMICAL, MIDLAND, MICHIGAN, SEPTEMBER 1984

Stated in Terms of Concentration (pg/m^3) on
Glass Fiber Filter/Polyurethane Foam (PUF) Plug

Date	Site	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
9/8-9/84													
	Site 1	ND/ND	0.30/ 0.69	ND/ND	0.95/ND	0.81/ND	1.15/*	ND/ND	ND/0.86	ND/ND	ND/ND	ND/ND	ND/ND
Average wind 199°, 6 mph	Site 2	ND/ND	4.82/39.98	1.95/7.33	ND/ND	2.08/ND	6.23/1.47	ND/ND	59.87/219.93	10.75/18.33	2.81/1.34	5.01/ND	3.42/ND
	Site 3	ND/ND	0.67/ 1.73	ND/ND	ND/ND	2.07/ND	7.31/0.61	ND/ND	9.98/ 4.74	0.54/ 3.90	ND/ND	ND/ND	0.57/0.79
	Site 4	ND/ND	0.14/ 0.72	ND/ND	0.86/ND	1.00/ND	2.69/ND	ND/ND	1.53/M	1.16/ND	ND/ND	ND/ND	1.66/ND
	Site 4 duplicate	ND/ND	0.06/ 0.42	ND/ND	ND/ND	1.54/ND	4.10/ND	ND/M	1.18/ 1.52	ND/ 1.41	0.73/ND	ND/ND	0.84/ND
9-12-13/84													
	Site 1	ND/ND	0.13/*	ND/ND	ND/ND	0.69/ND	1.66/ND	ND/ND	7.13/ 7.39	ND/ND	ND/ND	ND/ND	0.99/ND
Average wind 191°, 6 mph	Site 2	ND/ND	*/*	ND/ND	ND/ND	ND/ND	ND/ND	ND/ND	3.51/ 11.02	ND/ND	ND/ND	ND/ND	ND/ND
	Site 3	ND/ND	*/ 3.27	ND/ND	ND/ND	0.65/ND	5.10/ND	ND/ND	7.06/ 37.89	ND/ 2.22	ND/ND	ND/ND	0.81/ND
	Site 4	ND/ND	0.38/*	ND/ND	ND/2.93	0.22/1.26	2.67/4.08	ND/ND	2.80/ 11.08	0.74/ 0.32	ND/ND	ND/ND	ND/2.67
	Site 4 duplicate	ND/ND	*/*	ND/ND	ND/ND	0.48/ND	5.60/ND	ND/ND	2.55/ 8.66	1.69/ 1.32	ND/ND	ND/ND	ND/ND
9/22-23/84													
	Site.1	ND/ND	*/*	ND/ND	ND/ND	ND/ND	ND/0.30	ND/ND	2.22/ 0.22	ND/ND	ND/ND	ND/ND	ND/0.13
Average wind 212°, 5 mph	Site 2	ND/ND	0.49/21.86	ND/ND	0.55/ND	2.69/ND	7.57/6.72	ND/ND	65.33/ 90.36	2.69/ 4.76	4.52/ND	2.93/ND	1.34/0.26
	Site 3	ND/ND	*/ 0.59	ND/ND	ND/ND	0.55/ND	2.73/ND	ND/ND	4.35/ 7.25	ND/ND	ND/ND	ND/ND	ND/0.70
	Site 4	ND/ND	10.28/63.79	ND/1.37	0.28/ND	1.14/ND	4.01/ND	ND/ND	84.60/290.77	7.90/28.83	3.00/ND	3.00/ND	4.64/ND
	Site 4 duplicate	ND/M	24.28/M	ND/ND	0.96/ND	1.41/ND	4.37/ND	ND/ND	122.70/*	15.42/ND	4.37/ND	2.70/ND	6.55/ND

Keys to Symbols

ND-Not found at detection limit (example detection limit ranges: 0.05-0.62 pg/m^3 for 2378-TCDF, 0.03-1.62 pg/m^3 for 2378-TCDD)
M-Data not provided by analytical laboratory.
*-Exposed sample concentration lower than that of field blank-consider equivalent to nondetectable.

2. Semi-Volatile Compounds

Because of the large number of individual samples and compounds detected in sampling for semi-volatile compounds, it was decided to limit the full review of these data to those sampling periods in which consistently favorable relationships existed between monitoring sites upwind and downwind of Dow Chemical. Nine of the 18 sampling days were evaluated, with southerly to southwesterly winds having been present in eight of those nine days. These data are presented in Table VI-7.

Review of these data by the EPA Region V Central Regional Laboratory yielded the following principal findings:

1. Limited sampling media blank samples were analyzed. A polyurethane foam blank was found free of contamination. However, method blanks of XAD-2 resin contained measurable phenol; biphenyl; 2,4-dichlorophenol; 1,2,4-trichlorobenzene; tetrachlorobenzene, and 2-hydroxybenzaldehyde.

2. Field bias blanks frequently contained phenol, biphenyl, and diphenyl ether. These were subtracted from the quantities detected in field samples, as a correction.

3. Problems were observed with interferences or mass spectrum assignment criteria in some analyses for phenol and biphenyl. These data are labeled appropriately in Table VI-7.

4. Recoveries of acid and base-neutral surrogate compounds were generally not within acceptable limits. According to current guidance available concerning the interpretation of data affected in this way (see Section V.D. of this report), there is no agreed method to judge acceptability of compound-by-compound analytical data based on the recovery of specific surrogates. The semi-volatile compound data presented herein should be used in that context.

Nonetheless, positive identifications of many semi-volatile compounds were achieved, and higher concentrations of several semi-volatile compounds were found at sites downwind of Dow Chemical. For the data reviewed, precision, compound-by-compound (sample and field duplicate sample from site 4) was within target criteria for all detected compounds (± 50% RPD) on four days and the goal was nearly met on a fifth day. Significantly higher concentrations of most compounds including

1,2,3-trichlorobenzene
1,2,4-trichlorobenzene
1,3,5-trichlorobenzene
1,2,3,4-tetrachlorobenzene
1,2,4,5-tetrachlorobenzene
phenol
2,4-dichlorophenol
2,4,6-trichlorophenol
biphenyl, and
diphenyl ether (1,1-oxybisbenzene)

TABLE VI-7

RESULTS OF AMBIENT AIR SAMPLING FOR SEMI-VOLATILE COMPOUNDS
IN VICINITY OF DOW CHEMICAL COMPANY, MIDLAND, MICHIGAN, SEPTEMBER 1984
(Concentration ng/m^3)

Sampling Period	Site	Average Wind Direction and Speed	1,2,3-Trichlorobenzene	1,2,4-Trichlorobenzene	1,3,5-Trichlorobenzene	1,2,3,4-Tetrachloro-benzene	1,2,4,5-Tetrachloro-benzene	Pentachlorobenzene	Hexachlorobenzene	Phenol	2-Chlorophenol	3-Chlorophenol	2,4-Dichlorophenol	2,3,5-Trichlorophenol	2,4,5-Trichlorophenol	2,4,6-Trichlorophenol	Pentachlorophenol	2-Phenylphenol	4-Phenylphenol	Biphenyl	2-Hydroxybenzaldehyde	4-Hydroxybenzaldehyde	Diphenylether
9/7-8/84	1	184°, 5.9 mph	102	699	ND (21.2)	296	97.4	ND (21.2)	ND (21.2)	ND (21.2)	ND (21.2)	ND (21.2)	1693	ND (21.2)	ND (21.2)	78.3	ND (42.3)	ND (55.0)	ND (21.2)	ND (21.2)	ND (21.2)	ND (275)	1204
	2		50.3	402	ND (16.8)	184	62.0	15.1*	ND (16.8)	1546	ND (16.8)	ND (16.8)	35.2	ND (16.8)	41.9	ND (16.8)	ND (33.5)	117	ND (16.8)	109	ND (16.8)	ND (16.8)	1120
	3		ND (14.8)	26.6	ND (14.8)	16.2	ND (14.8)	ND (14.8)	ND (14.8)	181	ND (14.8)	ND (14.8)	ND (14.8)	ND (14.8)	ND (14.8)	ND (14.8)	ND (29.5)	ND (14.8)	ND (14.8)	249	19.2	ND (14.8)	161
	4				(Sample not analyzed.)																		
	4FD		ND (16.2)	29.2	ND (16.2)	11.4*[1]	4.86[1]	ND (16.2)	ND (16.2)	492[1]	ND (16.2)	ND (34.1)	ND (16.2)	ND (16.2)	ND (16.2)	ND (16.2)	ND (32.5)	ND (16.2)	1.6*[1]	ND (16.2)	ND (40.6)	ND (16.2)	117[1]
		Precision			(Site 4 sample not analyzed - precision not calculated.)																		
		Detected in blank								*										*			*
9/8-9/84	1	199°, 6.2 mph	ND (23.0)	ND (23.0)	ND (23.0)	ND (23.0)	ND (23.0)	ND (23.0)	ND (23.0)	ND (115)	ND (23.0)	ND (23.0)	ND (23.0)	ND (23.0)	ND (23.0)	ND (23.0)	ND (46.0)	ND (23.0)	ND (23.0)	36.8	ND (23.0)	ND (23.0)	(2)
	2		41.0	312	ND (16.4)	246	67.3	11.5*	ND (16.4)	128	4.92*	ND (16.4)	ND (16.4)	ND (16.4)	123	ND (72.2)	ND (32.8)	ND (16.4)	ND (16.4)	93.5	ND (16.4)	ND (16.4)	745
	3		30.2	146	ND (15.9)	70.0	49.4	ND (15.9)	ND (15.9)	1000	ND (15.9)	ND (15.9)	28.7[1]	ND (15.9)	23.9[1]	ND (15.9)	ND (31.8)	191	ND (15.9)	97.1	ND (15.9)	ND (15.9)	627
	4		ND (17.1)	23.9	ND (17.1)	13.7*	ND (17.1)	ND (17.1)	ND (17.1)	389	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (34.1)	ND (17.1)	ND (17.1)	35.8	ND (17.1)	ND (17.1)	17.1
	4FD		ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	(2)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (34.1)	ND (17.1)	ND (17.1)	1.71*	ND (17.1)	ND (17.1)	ND (17.1)
		Precision		--						--										181.8			--
		Detected in blank								*										*			*
9/12-13/84	1	191°, 5.6 mph	ND (17.0)	5.09*	ND (17.0)	ND (5.1)*	ND (17.0)	ND (17.0)	ND (17.0)	84.9	ND (17.0)	ND (17.0)	ND (17.0)	ND (17.0)	ND (17.0)	ND (17.0)	ND (34.0)	3.40*	6.79*	35.7	ND (17.0)	ND (17.0)	ND (17.0)
	2		ND (17.9)	250	1.79*	ND (17.9)	55.5	ND (17.9)	ND (17.9)	ND (17.9)	ND (17.9)	ND (17.9)	4114	ND (17.9)	172	ND (17.9)	ND (35.8)	136	ND (17.9)	94.8	ND (17.9)	37.6	1789
	3		37.3	258	ND (31.1)	115	46.6	ND (31.1)	ND (31.1)	(2)	ND (31.1)	ND (31.1)	1398	ND (31.1)	ND (31.1)	ND (31.1)	ND (62.1)	ND (31.1)	ND (31.1)	ND (31.1)	ND (31.1)	74.5	1025
	4		108	855	ND (37.2)	409	141	14.9*	ND (37.2)	770	ND (37.2)	ND (37.2)	2232	ND (37.2)	ND (37.2)	ND (37.2)	ND (74.4)	78.1	ND (37.2)	338	ND (37.2)	ND (37.2)	2194
	4FD		ND (27.4)	ND (27.4)	ND (27.4)	ND (27.4)	ND (27.4)	ND (27.4)	ND (27.4)	35.6	ND (27.4)	ND (27.4)	ND (27.4)	ND (27.4)	ND (27.4)	ND (27.4)	ND (54.8)	ND (27.4)	(27.4)	27.4	(27.4)	(27.4)	16.4*
		Precision								182.3										172.8			197.0
		Detected in blank								*										*			

TABLE VI-7 (continued)
(Concentration ng/m^{3})

Sampling Period	Site	Average Wind Direction and Speed	1,2,3-Trichlorobenzene	1,2,4-Trichlorobenzene	1,3,5-Trichlorobenzene	1,2,3,4-Tetrachloro-benzene	1,2,4,5-Tetrachloro-benzene	Pentachlorobenzene	Hexachlorobenzene	Phenol	2-Chlorophenol	3-Chlorophenol	2,4-Dichlorophenol	2,3,5-Trichlorophenol	2,4,5-Trichlorophenol	2,4,6-Trichlorophenol	Pentachlorophenol	2-Phenylphenol	4-Phenylphenol	Biphenyl	2-Hydroxybenzaldehyde	4-Hydroxybenzaldehyde	Diphenylether
9/14-15/84	1	331°, 6.6 mph	ND (15.9)	20.6	ND (15.9)	7.94*	ND (15.9)	ND (15.9)	ND (15.9)	38.1	ND (15.9)	ND (15.9)	ND (15.9)	ND (15.9)	ND (15.9)	ND (15.9)	ND (31.7)	ND (15.9)	ND (15.9)	28.6	ND (15.9)	ND (15.9)	ND (15.9)
	2		ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	162	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	ND (21.0)	18.9	ND (21.0)	ND (21.0)	23.1*
	3		ND (14.6)	ND (14.6)	ND (14.6)	ND (14.6)	ND (14.6)	ND (14.6)	ND (14.6)	33.6	ND (14.6)	ND (14.6)	ND (14.6)	ND (14.6)	ND (14.6)	ND (14.6)	ND (29.2)	ND (14.6)	ND (14.6)	17.5	ND (14.6)	ND (14.6)	ND (14.6)
	4		ND (13.8)	ND (13.8)	ND (13.8)	ND (13.8)	ND (13.8)	ND (13.8)	ND (13.8)	83.0	ND (13.8)	ND (13.8)	ND (13.8)	ND (13.8)	ND (13.8)	ND (13.8)	ND (27.7)	ND (13.8)	ND (13.8)	19.4	ND (13.8)	ND (13.8)	ND (13.8)
	4FD		ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (37.6)	ND (75.1)	ND (37.6)	ND (37.6)	26.3	ND (37.6)	ND (37.6)	ND (37.6)
		Precision								84.7										30.2			
		Detected in blank																					
9/17-18/84	1	212°, 4.1 mph	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (16.0)	ND (32.0)	ND (16.0)	ND (16.0)	36.8	4.8*	ND (16.0)	3.2*
	2		43.0	362	10.3*	327	126	ND (17.2)	ND (17.2)	671	ND (17.2)	ND (17.2)	112	534	ND (17.2)	189	ND (34.4)	ND (17.2)	ND (17.2)	122	ND (17.2)	ND (17.2)	ND (17.2)
	3		10.1*	37.4	ND (14.4)	ND* (21.6)	12.9*	ND (14.4)	ND (14.4)	ND (14.4)	ND (14.4)	ND (14.4)	ND (14.4)	ND (14.4)	ND (14.4)	ND (14.4)	ND (28.8)	ND (24.4)	ND (14.4)	104	7.19*	ND (14.4)	575
	4		41.7	233	ND (16.7)	167	60.0	5.0*	ND (16.7)	112	ND (16.7)	ND (16.7)	23.3	ND (16.7)	ND (16.7)	ND (16.7)	ND (33.3)	5.0*	ND (16.7)	53.3	ND (16.7)	ND (16.7)	283
	4FD		31.1	173	ND (17.3)	91.5	32.8	3.45*	ND (17.3)	259	ND (17.3)	ND (17.3)	ND (17.3)	ND (17.3)	ND (17.3)	ND (17.3)	ND (34.5)	ND (17.3)	ND (17.3)	ND (17.3)	ND (17.3)	ND (17.3)	157
		Precision	29.1	29.6		56.1	52.1	36.7		79.2			--					--		--			57.3
		Detected in blank																		•			
9/18-19/84	1	235°, 4.0 mph	ND (22.5)	ND (22.5)	ND (22.5)	ND (22.5)	ND (22.5)	ND (22.5)	ND (22.5)	405	ND (22.5)	ND (22.5)	ND (22.5)	ND (22.5)	ND (22.5)	ND (22.5)	ND (45.0)	ND (22.5)	ND (22.5)	67.6	22.5	ND (22.5)	ND (22.5)
	2		130	566	ND (17.1)	823	326	ND (17.1)	ND (17.1)	1715	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (17.1)	ND (34.2)	ND (17.1)	ND (17.1)	92.6	ND (17.1)	ND (17.1)	2227
	3		ND (17.2)	ND (17.2)	ND (17.2)	ND (17.2)	ND (17.2)	ND (17.2)	141	ND (17.2)	ND (17.2)	ND (17.2)	ND (17.2)	ND (17.2)	ND (17.2)	ND (17.2)	ND (34.5)	ND (17.2)	ND (17.2)	22.4*^{3}	ND (17.2)	ND (17.2)	ND (17.2)
	4		62.6	457	10.2*	237	110	8.5*	ND (16.9)	812	ND (16.9)	ND (16.9)	2538	ND (16.9)	ND (16.9)	127	ND (33.8)	40.6	3.38*	112*^{3}	ND (16.9)	ND (16.9)	1149
	4FD		54.4	429	10.0*	215	97.3	ND (14.3)	ND (14.3)	601	ND (14.3)	ND (14.3)	2003	ND (14.3)	ND (14.3)	104	ND (28.6)	ND (14.3)	ND (14.3)	104*^{3}	ND (14.3)	ND (14.3)	1086
		Precision	14.0	6.3		9.7	12.3	--		29.9			23.6			19.9		--		7.4			5.6
		Detected in blank																		•			•

TABLE VI-7 (continued)
(Concentration ng/m^3)

Sampling Period	Site	Average Wind Direction and Speed	1,2,3-Trichlorobenzene	1,2,4-Trichlorobenzene	1,3,5-Trichlorobenzene	1,2,3,4-Tetrachloro-benzene	1,2,4,5-Tetrachloro-benzene	Pentachlorobenzene	Hexachlorobenzene	Phenol	2-Chlorophenol	3-Chlorophenol	2,4-Dichlorophenol	2,3,6-Trichlorophenol	2,4,5-Trichlorophenol	2,4,6-Trichlorophenol	Pentachlorophenol	2-Phenylphenol	4-Phenylphenol	Biphenyl	2-Hydroxybenzaldehyde	4-Hydroxybenzaldehyde	Diphenylether
9/22-23/84	1	212°, 4.9 mph	(Incomplete sampling run -- sample not analyzed.)																				
	2		57.0	326	ND (16.3)	151	83.0	ND (16.3)	ND (16.3)	6941	ND (16.3)	ND (16.3)	ND (16.3)	ND (16.3)	ND (16.3)	179	ND (32.6)	ND (16.3)	ND (16.3)	111	ND (16.3)	ND (16.3)	1530
	3		7.16*	28.6	ND (14.3)	21.5	18.6	ND (14.3)	ND (14.3)	1379	ND (14.3)	ND (14.3)	ND (14.3)	ND (14.3)	ND (14.3)	ND (14.3)	ND (28.6)	ND (14.3)	ND (14.3)	80.2*[3]	ND (14.3)	ND (14.3)	286
	4		61.0	425	3.70*	296	106	11.1*	ND (18.5)	956	ND (18.5)	ND (18.5)	721	ND (18.5)	ND (18.5)	181	ND (37.0)	ND (18.5)	ND (18.5)	115*[3]	ND (18.5)	ND (18.5)	1128
	4FD		52.8	347	3.30*	314	107	ND (16.5)	ND (16.5)	649	ND (16.5)	ND (16.5)	528	ND (16.5)	ND (16.5)	162	ND (33.0)	ND (16.5)	ND (16.5)	129*[3]	ND (16.5)	ND (16.5)	1090
		Precision	14.4	20.2	11.4	5.9	0.0			38.3			30.9			11.1				11.5			3.4
		Detected in blank								•										•			
9/23-24/84	1	197°, 2.6 mph	3.23*	9.21*	ND (16.2)	21.0	3.24*	ND (16.2)	ND (16.2)	359	ND (16.2)	ND (16.2)	ND (16.2)	ND (16.2)	ND (16.2)	ND (16.2)	ND (32.4)	ND (16.2)	ND (16.2)	64.7*[3]	53.4	ND (16.2)	ND (16.2)
	2		30.8	199	ND (18.1)	181	77.8	ND (18.1)	ND (18.1)	6666	ND (18.1)	ND (18.1)	959	ND (18.1)	ND (18.1)	ND (18.1)	ND (36.2)	81.5	ND (18.1)	170[3]	ND (18.1)	ND (18.1)	1792
	3		10.2*	67.9	ND (17.0)	52.6	25.4	ND (17.0)	ND (17.0)	1836	ND (17.0)	ND (17.0)	ND (17.0)	ND (17.0)	ND (17.0)	30.5	ND (33.9)	ND (17.0)	ND (17.0)	96.7	ND (17.0)	ND (17.0)	594
	4		14.7*	101	ND (18.3)	75.2	25.7	ND (18.3)	ND (18.3)	224	ND (18.3)	ND (18.3)	ND (18.3)	ND (18.3)	ND (18.3)	25.7	ND (36.7)	ND (18.3)	ND (18.3)	95.3[3]	ND (18.3)	ND (18.3)	440
	4FD		12.3*	91.7	ND (17.6)	68.8	24.7	1.76*	ND (18.3)	215	ND (17.6)	ND (17.6)	ND (17.6)	ND (17.6)	ND (17.6)	24.7	ND (35.3)	ND (17.6)	ND (17.6)	95.2	ND (17.6)	ND (17.6)	459
		Precision	17.8	9.7		8.9	4.0	--		4.1						4.0				0.1			4.2
		Detected in blank								•													
9/24-25/84	1	195°, 4.9 mph	5.57*	13.0*	ND (18.6)	24.1	ND (18.6)	ND (18.6)	ND (18.6)	186	ND (18.6)	ND (18.6)	ND (18.6)	ND (18.6)	ND (18.6)	ND (18.6)	ND (37.1)	ND (18.6)	ND (18.6)	16.7	ND (18.6)	ND (18.6)	3.7*
	2		38.8	254	ND (18.5)	203	83.1	ND (18.5)	ND (18.5)	1736[h]	ND (18.5)	ND (18.5)	ND (18.5)	ND (18.5)	ND (18.5)	127	ND (36.9)	ND (18.5)	(18.5)	122	(18.5)	(18.5)	720
	3		14.2	65.3	ND (14.2)	75.3	41.2	2.8*	ND (14.2)	1250	ND (14.2)	ND (14.2)	ND (14.2)	ND (14.2)	ND (14.2)	31.2	ND (28.4)	63.9	ND (14.2)	115	19.9	ND (14.2)	469
	4		6.46*	51.7	ND (16.1)	43.6	24.2	ND (16.1)	ND (16.1)	436[h]	ND (16.1)	ND (16.1)	ND (16.1)	ND (16.1)	ND (16.1)	ND (16.1)	ND (32.3)	ND (16.1)	ND (16.1)	51.7[3]	ND (16.1)	ND (16.1)	194
	4FD		10.8*	77.1	ND (17.9)	62.8	37.7	ND (17.9)	ND (17.9)	508[h]	ND (17.9)	ND (17.9)	ND (17.9)	ND (17.9)	ND (17.9)	ND (17.9)	ND (35.9)	ND (17.9)	ND (17.9)	68.2	ND (17.9)	ND (17.9)	251
		Precision	50.3	39.4		36.1	43.6			22.4										27.5			25.6
		Detected in blank																					

TABLE VI-7 (continued)

NOTES: ND = Not detected.
FD = Field duplicate sample.
[*] = Estimated value.
[1] = Identification and quantitation of dichlorophenol and trichlorophenol suspect.
[2] = Concentration in blank higher than in sample.
[3] = Quantitation of biphenyl suspect; all mass spectrum assignment criteria were not met.
[4] = Interferences present in mass spectrum; suspected positive bias.

were detected principally at downwind monitors, in the following sampling periods:

9/18-19/84
9/22-23/84
9/23-24/84, and
9/24-25/84

with precision achieving the target criterion for many compounds on 9/17-18/84. As indicated above, southerly to southwesterly wind patterns were considered most appropriate to judge upwind-downwind relationships. As a control, one sampling period in which northerly winds were present, 9/14-15/84, was reviewed to determine whether any of the compounds were present when little or no wind contacted the Dow Chemical facility prior to collection in the samplers. As expected, most compounds were not detected. These data demonstrate that the Dow Chemical facility does emit measurable quantities of semi-volatile compounds. In addition, the range of tentatively identified compounds (see Table VI-8) was generally larger at downwind monitoring sites.

Referring to Table V-15 (Section V of this report), the identifiable semi-volatile compounds measured in Building 703 incinerator exhaust were few, and were in the range of 10 to 100 ppb. The complement of semi-volatile compounds presented in Tables VI-7 and VI-9 is much more extensive; further, the single compound detected in both the incinerator exhaust and ambient air sampling, tetrachlorobenzene, was found to be present in ambient air at a level considerably higher than expected if the incinerator exhaust were the sole source. Applying an approximate dilution factor of 10^5 to account for the distance and elevation difference of the ambient monitoring sites with respect to the incinerator stack, to the tetrachlorobenzene concentration presented in Table V-15, an approximate ground level concentration of tetrachlorobenzene (1,2,3,4 plus 1,2,4,5 isomer) would be in the range of 0.1 ppt rather than the maximum concentrations between 100 and 1000 ppt shown in Table VI-9. These data suggest that sources within the Dow Chemical facility other than the Building 703 incinerator exhaust stack, such as process vents or fugitive emissions sources, may be attributable for the levels of semi-volatile compounds detected in ambient air around the plant. It is known that 2,4-dichlorophenol is currently produced at the Midland plant. The finding of 2,4,5-trichlorophenol is surprising in that Dow Chemical has not produced 2,4,5-trichlorophenol for some time, nor does the company report any current use of 2,4,5-trichlorophenol to any significant extent.[23]

3. Volatile Compounds

As described previously, these compounds were sampled using traps packed with carbon molecular sieves (CMS) and a study was conducted to demonstrate the validity of this sorbent for the compounds to be sampled. Most of the CMS tubes used in the validation study were not analyzed by the contract laboratory within required times; results from those tubes analyzed before their expiration are shown in Table VI-10. Seven of the eight compounds spiked into the tubes were not detected. The detection of perchloroethylene (tetrachloroethylene), the remaining spiked compound, was not in consistent agreement with the known levels spiked.

TABLE VI-8

TENTATIVELY IDENTIFIED SEMI-VOLATILE COMPOUNDS DETECTED IN AMBIENT AIR SAMPLING IN VICINITY OF DOW CHEMICAL COMPANY, MIDLAND, MICHIGAN, SEPTEMBER 1984

Sampling Period	Site	Average Wind Direction and Speed	Compounds Tentatively Identified
9/7-8/84	1	184°, 6 mph	Ethylcyclopentane; methylcyclohexane; xylene; methylethylbenzene; dichlorobenzene; methylnaphthalene
	2		Methylnaphthalene; dichlorobenzene; benzoic acid; 1,2-diethylbenzene; ethenylmethylbenzene; ethylmethylbenzene; ethylmethylbenzene; chlorobenzene; toluene
	3		Methylphenanthrene; phenylbicyclohexyl; terphenyl; methylnaphthalene; bis(dimethylethyl)phenol; xylene; toluene
	4		(Sample not analyzed.)
	4FD		Ethylbenzene; ethylmethylbenzene; biphenyl
	4FB		Naphthalene; ethylmethylbenzene; toluene; ethylcyclopentane
9/8-9/84	1	199°, 6 mph	Ethylmethylbenzene; propylbenzene; toluene; benzene; benzothiazole; xylene
	2		Diethylbenzene; ethenylethylbenzene; propylbenzene; ethylmethylbenzene; ethylbenzene; toluene
	3		Dichlorobenzene; diethylbenzene; trimethylnaphthalene; chlorobenzene; dimethylbenzene; styrene; ethylmethylbenzene; ethenylethylbenzene; diethenylbenzene; methylbenzaldehyde; ethylbenzene
	4		Diethylbenzene; ethylbenzene; ethenylbenzene; ethylmethylbenzene; 2,3-dihydroindene; ethenylethylbenzene; diethenylbenzene
	4FD		Ethylbenzene; ethenylbenzene; ethylmethylbenzene
	4FB		Ethylmethylbenzene; trimethylbenzene; benzene; ethylbenzene; diethylbenzene
9/12-13/84	1	191°, 6 mph	Hexanedioic acid dioctyl ester; dodecanonitrile; di-1,2-benzenedicarboxylic acid; ethylbenzoic acid; 2,6-bis (1,1-dimethyl)phenol; 1,1'-(1,4-phenylene) B ethanone; benzoic acid; 1,2,3-trimethylbenzene, 1-ethyl-2-methylbenzene; ethylbenzene; xylene; acetic acid butylester
	2		1-Methylethylbenzene; 2,3-dihydro 1 H-indene; 1,3-diethylbenzene, 1-ethenyl-4-ethylbenzene; 1,1'-oxybisbenzene; hexadecanoic acid methylester; 2-methylnaphthalene
	3		3,7-Dimethyl-1,6-octadien-3-ol; 1-ethyl-2-methylbenzene; 1-methylethylbenzene; xylene; 2,2-dimethyloctanol; bis(2-ethylhexyl) hexanedioic acid; methylethylbenzene; ethylbenzene; 1,2-diethylbenzene; xylene; 1,4-dihydro-1,4-methanonaphthalene
	4		Diethylbenzene; ethylbenzene; xylene; 2,3-dihydro 1 H-indene; 1-methylethylbenzene
	4FD		1-Methylethylbenzene; 2-ethylhexanoic acid; 2,6-bis(1,1-dimethylethyl)phenol; 4-methyl-1,3-benzenediamine; 5,7-methylundecane; 2-cyclohexen-1-one
	4FB		Dimethylbenzene; bicyclo [4.2.0] octa-1,3,5-triene; 1,3,6-octatriene; 3,7-dimethyl; 1-ethyl-2-methylbenzene; 6,6-dime bicyclo [3.1.1] heptane; octamethylcyclotetrasiloxane; dodecamethylcyclohexasiloxane; 2-methyltridecane; di-1,2-benzene dicarboxylic acid; 2,10-methylundecane; 2,6-bis(1,1-dimethyl) phenol; 5,7-dimethylundecane; 2-fluorophenol; ethylbenzene; hexanedioic acid dioctylester; 2,7-dimethyloctane; 1-nitroethylbenzene
9/14-15/84	1	331°, 7 mph	Xylene; 4-methyl-1-(3)-cyclohexen-1-ol; 1-ethyl-2-methylbenzene; ethylmethylbenzene; hexadecanoic acid; ethylbenzene methylethylbenzene
	2		Xylene; ethylmethylbenzene; 1,2-diethylbenzene; 1,2,3,4-tetramethylbenzene; 1-ethylnaphthalene; hexadecanoic acid methylester; methylbenzene; ethylmethylbenzene; 1,2,4-trimethylbenzene; 1-ethyl-2,3-dimethylbenzene; methylnaphthalene; 1,4-dihydro-1,4-methanonaphthalene
	3		3-Bromodecane; hexadecanoic acid dioctylester; ethylmethylbenzene; xylene
	4		3-Bromodecane; ethylbenzene; 1-methylethylbenzene, 2-propylheptanol, xylene
	4FD		1-Acetyl-1,2,3,4-tetrapyridine; 3-bromodecane; ethyldimethylbenzene; 2-methylpropylbenzene; ethylmethylbenzene
	4FB		Methylcyclohexane; methylbenzene; di-1,2-benzenedicarboxylic acid; 2-propenylindenocyclobutene; methylethylbenzene

TABLE VI-8 (continued)

Sampling Period	Site	Average Wind Direction and Speed	Compounds Tentatively Identified
9/17-18/84	1	212°, 4 mph	2-Methylnaphthalene; 1,1-dimethylethylbenzene; 4-ethyl-1,2-dimethylbenzene; 1,2,3,4-tetramethylbenzene; ethylbenzene; benzoic acid; 1-methyl-4-propylbenzene; 2-methylnaphthalene; ethylcyclopentane; 11-nitro-1-undecane; dimethylbenzene; 1,3,5-cycloheptatriene; 4-methyl-1,3-cyclohexen-1-ol; ethylmethylbenzene; trimethylbenzene; ethyldimethylbenzene; 1,3-dimethylbenzene
	2		2,4-Hexadiyne; methylbenzene; xylene; 1,3,5,7-cyclooctatetraene; 1-methylethylbenzene; diethylbenzene; undecane; naphthalene; methylnaphthalene; 3,4,5-trimethylhexene; dimethylpentene; 4-ethenylcyclohexene; chlorobenzene; ethylbenzene
	3		Methylnaphthalene; benzenedicarbonitrile; 1,1-dimethylethylbenzene; 1-methylpropylbenzene; 1,2,4-trimethylbenzene; 1-methylethylbenzene; 1-ethyl-2-methylbenzene; xylene; chlorobenzene; methylcyclohexane; 1,2-dimethyl-4-ethylbenzene; 2-methyldecahydronaphthalene; propylbenzene
	4		Ethylbenzene; 4-(6-methyl-2-benz) benzamine; 1-undecane, 11-nitro; o,o-diethylphosphorothio acid; 2-propyl-1-heptanol; 1,2-benzenedicarbonitrile; xylene; 1,3,5,7-cyclooctatetraene
	4FD		2-Methylphenanthrene; 1-(2-bromoethyl)-3-fluorobenzene; 2,4-dinitrobenzeneamine; 2,2,7,7-tetra-4,5-octadien-3-one; 2-ethyl-2H-benzotriazole; 1,1'-(1,4-phenylene) B-ethanone methylsulfonylbenzene; dichlorobenzene; 1-ethyl-4-methylbenzene; 1-methylethenylbenzene; ethylbenzene; 1,2-diethylbenzene; 1-methylethylbenzene; xylene
	4FB		1,2,3-Trimethylbenzene; di-1,2-benzenedicarboxylic acid; 1-methylethylbenzene; methylbenzene; 1-ethyl-2-methylbenzene
9/18-19/84	1	235°, 4 mph	Dimethylbenzene; ethylmethylbenzene; methylbropylbenzene, trimethylbenzene; benzenedicarbonitrile
	2		Chlorobenzene; ethylbenzene; ethenylbenzene; methylethylbenzene; dichlorobenzene; diethylbenzene; methylbenzofuran
	3		Pyrene; dimethylbenzene; ethylbenzene; ethyldimethylbenzene; methylethylbenzene; propylbenzene; methylpropylbenzene
	4		Dimethylbenzaldehyde; dimethylbenzene; dichlorobenzene; ethylmethylbenzene; ethenylbenzene; methylbenzaldehyde; diethenylbenzene
	4FD		Methylbenzaldehyde; diethylbenzene; ethylmethylbenzene; dimethylbenzene; dichlorobenzene; ethenylbenzene; ethylbenzene
	4FB		Dimethylbenzaldehyde; diethenylbenzene; ethenylethylbenzene; diethenylbenzene
9/22-23/84	1	212°, 5 mph	1,1'-(1,4-Phenylene)bis ethanone; benzoic acid; ethylmethylbenzene; dimethylbenzene
	2		Ethylbenzene; dimethylethylbenzene; phenanthrene, diethylbenzene; dichlorobenzene; ethenylbenzene; chlorobenzene
	3		Ethylmethylbenzene; dichlorobenzene; diethylbenzene, ethyldimethylbenzene; methylnaphthalene; naphthalene; diethenylbenzene; methylpropylbenzene; dimethylbenzene
	4		Methylethylbenzene; diethylbenzene; ethylbenzene
	4FD		Ethenylethylbenzene; diethylbenzene; methylethylbenzene; ethenylbenzene; ethylbenzene; diethenylbenzene; dichlorobenzene; dimethylbenzene
	4FB		Dimethylbenzaldehyde; propylbenzene; ethylmethylbenzene
9/23-24/84	1	197°, 3 mph	Methylnaphthalene; naphthalene; dimethylethylbenzene; methylpropylbenzene; methylethylbenzene; dimethylbenzene
	2		Anthracene; methylethylbenzene; ethenylbenzene; dimethylbenzene; dichlorobenzene; ethylbenzene; diethylbenzene; 1,1'-(1,4-phenylene)bis ethanone; naphthalene; diethenylbenzene
	3		Diethylbenzene; ethenylethylbenzene; ethylbenzene; 1,1'-(1,4-phenylene)bis ethanone; methylnaphthalene; naphthalene; ethenylethylbenzene; dichlorobenzene; ethenylbenzene; dimethylbenzene
	4		1,1'-(1,4-Phenylene)bis ethanone; dimethylbenzene; ethenylethylbenzene; diethenylbenzene; ethyldimethylbenzene; diethylbenzene; ethylbenzene; 1,1'-oxybisbenzene; naphthalene; dichlorobenzene
	4FD		Dimethylnaphthalene; naphthalene; ethylbenzene; methylethylbenzene; propylbenzene; diethenylbenzene; dimethylethylbenzene; diethylbenzene; dimethylbenzene; dichlorobenzene; ethenylethylbenzene
	4FB		Propylbenzene; trimethylbenzene; methylethylbenzene; dimethylbenzene

TABLE VI-8 (continued)

Sampling Period	Site	Average Wind Direction and Speed	Compounds Tentatively Identified
9/24-25/84	1	195°, 5 mph	1,1'-(1,4-Phenylene)bis ethanone; methylethylbenzene; dimethylbenzene
	2		Ethylbenzene; methylethylbenzene; diethylbenzene; diethenylbenzene; naphthalene; 1,1'-oxybisbenzene; methylbenzene; dimethylbenzene; dichlorobenzene; methylnaphthalene; 1,1'-(1,4-phenylene)bis ethanone
	3		Methylnaphthalene; diethylbenzene; methylethylbenzene; 1,1'-(1,4-phenylene)bis ethanone; diethenylbenzene
	4		Dimethylbenzene; ethylmethylbenzene; diethylbenzene; ethenylethylbenzene; diethenylbenzene; naphthalene; 1,1'-oxybisbenzene; tetramethylbenzene
	4FD		Dimethylbenzene; diethylbenzene; ethenylethylbenzene; dimethylbenzene; diethenylbenzene; methylphenylethanone; 1,1'-oxybisbenzene
	4FB		Diethylbenzene; methylethylbenzene; dimethylbenzaldehyde

NOTES: FD = Field duplicate sample.
FB = Field blank sample.

TABLE VI-9

RANGES OF CONCENTRATIONS OF QUANTITATED SEMI-VOLATILE COMPOUNDS IN AMBIENT AIR ON NINE SAMPLING DAYS - MIDLAND, MICHIGAN

9/7/84 - 9/25/84
(Data expressed in ng/m^3)

Site		1,2,3-Trichlorobenzene	1,2,4-Trichlorobenzene	1,3,5-Trichlorobenzene	1,2,3,4-Tetrachlorobenzene	1,2,4,5-Tetrachlorobenzene	Pentachlorobenzene	Hexachlorobenzene	Phenol	2-Chlorophenol	3-Chlorophenol	2,4-Dichlorophenol	2,3,5-Trichlorophenol	2,4,5-Trichlorophenol	2,4,6-Trichlorophenol	Pentachlorophenol	2-Phenylphenol	4-Phenylphenol	Biphenyl	2-Hydroxybenzaldehyde	4-Hydroxybenzaldehyde	Diphenylether
1	Maximum	102	699	ND	296	97.4	ND	ND	405	ND	ND	1693	ND	ND	78.3	ND	3.4	6.79	67.6	53.4	ND	1204
	Minimum	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Average	13.8	95.4	--	43.6	12.6	--	--	148	--	--	242	--	--	9.79	--	--	0.85	35.9	10.1	--	151
2	Maximum	130	566	10.3	327	326	15.1	ND	6941	4.92	ND	4114	534	172	189	ND	136	ND	170	ND	37.6	2227
	Minimum	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	18.9	ND	ND	ND
	Average	43.4	297	1.34	235	97.9	2.96	--	1507	0.55	--	580	59.3	37.4	60.6	--	37.2	--	104	--	4.18	1105
3	Maximum	37.3	258	ND	115	49.4	2.8	141	1836	ND	ND	1398	ND	239	31.2	ND	191	ND	249	19.9	74.5	1025
	Minimum	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Average	12.1	70.0	--	39.0	21.6	0.31	15.7	641	--	--	158	--	2.65	6.86	--	28.3	--	84.4	5.14	8.28	415
4	Maximum	108	855	10.2	409	141	14.9	ND	956	ND	ND	2538	ND	ND	181	ND	78.1	3.38	358	ND	ND	2194
	Minimum	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Average	26.8	194	1.60	118	45.4	2.63	--	385	--	--	473	--	--	36.7	--	7.28	0.29	93.8	--	--	498

TABLE VI-10

COMPARATIVE RESULTS OF CARBON MOLECULAR SIEVE TUBE VALIDATION STUDY

Tube Identification	Compound	Amount Spiked (ng)	Amount Detected (ng)
4-E	perchloroethylene	131	20.0
5-A	1,1,1-trichloroethane	(not spiked)	1290
	perchloroethylene	26	31.8
5-B	1,1,1-trichloroethane	(not spiked)	537
	perchloroethylene	26	56.5
5-C	(no spiked compounds detected)		

Also, because of difficulties relating primarily to sample holding times prior to analysis and possible blank contamination, most CMS tubes were not analyzed successfully. Therefore, the data for volatile compounds in ambient air presented in Table VI-11 are presented in qualitative terms.

From these data the following general conclusions appear supportable:

1. On each sampling day, site 1 was considered upwind of Dow Chemical. A wider range of compounds was usually detected at the downwind sites.

2. Two compounds, 1,1,1-trichloroethane and perchloroethylene, were found in most samples on the eight days for which analytical data are available. However, both compounds were frequently found as a blank contamination. Also, 1,1,1-trichloroethane appeared at high levels in the method validation study, though it was not spiked.

3. Precision between field duplicate samples was generally poor.

4. On each sampling day, either the low-flow or high-flow set of CMS tubes was designated the primary set for analysis, based upon ambient temperature and humidity conditions (see Appendix F, Section III.A). There was no distinct superiority or consistent pattern in the levels of compound detection in primary tubes.

5. Acrylonitrile and chloroform, when detected, were found primarily at monitoring sites downwind of Dow Chemical.

In addition to the six compounds appearing in Table VI-11, three compounds: monochlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene, were not detected in any sample. However, many of the volatile compounds selected for analysis (see Section II of this report) were not included. Among these compounds were benzene, ethylene dibromide, ethylene dichloride, ethylene oxide, methyl chloroform, methylene chloride, and vinylidene chloride. Several of these compounds were detected in Building 703 incinerator exhaust, as described in Section V of this report.

Thus, this portion of the ambient air study was not successful in scanning for the full range of desired compounds, either because of sampling or analytical method unsuitability, or insurmountable analytical problems. The available data should be considered qualitative.

4. Formaldehyde

The analytical results appearing in Table VI-12 show higher levels of formaldehyde in method and field blanks than in any of the 25 exposed field samples, with two exceptions. These data, evaluated by the EPA Region V Central Regional Laboratory as acceptable in terms of analytical accuracy, are not usable for quantifying the presence or absence of formaldehyde in ambient air during the study period.

TABLE VI-11

RESULTS OF AMBIENT AIR SAMPLING FOR VOLATILE COMPOUNDS IN VICINITY OF DOW CHEMICAL COMPANY, MIDLAND, MICHIGAN, SEPTEMBER 1984

Dates (1984)	Wind Direction and Average Speed	Site	High or Low Flow[1]	Acrylonitrile	Chloroform	1,2-dichloroethane	1,1,1-trichloroethane	Carbon tetrachloride	Perchloroethylene	Comments
9/7-8	184°, 6 mph	1	H						X*	
		2	L						X*	
		2	H				X*		X*	
		3	L		X		X		X*	
		3	H	X	X		X		X*	
9/8-9	199°, 6 mph	1	H				X*			
		2	L	X	X	X	X	X		
		2	H						X	
		3	L				X			
		3	H		X		X		X*	
		4	L				X		X	Duplicate sample only
9/12-13	191°, 6 mph	1	H				X		X*	
		2	L							
		2	H		X	X	X		X	
		3	L		X	X			X*	
		3	H			X			X*	
		4	H		X	X	X*			
9/17-18	212°, 4 mph	1	H		X		X	X	X	Field blank not analyzed
		2	L	X	X		X	X	X	
		3	H				X			
		4	L	X			X	X	X	
		4	H				X	X	X	
		4	H		X	X		X		Field duplicate sample

TABLE VI-11 (continued)

Dates (1984)	Wind Direction and Average Speed	Site	High of Low Flow[1]	Acrylonitrile	Chloroform	1,2-dichloroethane	1,1,1-trichloroethane	Carbon tetrachloride	Perchloroethylene	Comments
9/19-20	250°, 4 mph	1	H							
		2	L							
		3	H		X		X*			
		4	L						X	
		4	H		X		X*		X*	
		4	H	X	X		X	X	X	Field duplicate sample
9/22-23	212°, 5 mph	1	H				X	X		Field blank not analyzed
		2	L					X		
		2	H		X		X*			
		3	L					X		
		3	H							
		4	L							
		4	L				X			Field duplicate sample
9/23-24	197°, 3 mph	1	L							
		2	L				X			
		2	H	X	X		X	X		
		3	L				X		X	
		4	L							
		4	L				X			Field duplicate sample
9/24-25	195°, 5 mph	3	L				X	X		Field blank not analyzed
		4	L							

Notes: *Denotes compound detected at higher concentration in field blank sample.

[1] Primary tubes (high or low flow) are underlined in this category.

TABLE VI-12

RESULTS OF AMBIENT AIR SAMPLING FOR FORMALDEHYDE IN VICINITY OF DOW CHEMICAL COMPANY, MIDLAND, MICHIGAN, SEPTEMBER 1984

Date (1984)	Wind Direction and Speed (mph)	Sample Identification	Formaldehyde Derivative Detected (ug/sample)
9/7-8	184°, 6 mph	Method Blank	5.34
		Field Blank	4.78
		Site 1	2.04
		Site 2	4.09
		Site 3	2.68
		Site 4	1.89
		Site 4 Duplicate	2.15
9/8-9	199°, 6 mph	Method Blank	5.24
		Field Blank	3.91
		Site 1	1.13
		Site 2	1.69
		Site 3	1.00
		Site 4	0.95
		Site 4 Duplicate	0.79
9/12-13	191°, 6 mph	Method Blank	2.52
		Field Blank	2.11
		Site 1	1.46
		Site 2	0.14
		Site 3	0.19
		Site 4	0.29
		Site 4 Duplicate	0.22
9/18-19	235°, 4 mph	Method Blank	2.24
		Field Blank	1.80
		Site 1	0.51
		Site 2	0.90
		Site 3	0.55
		Site 4	0.36
		Site 4 Duplicate	2.91
9/19-20	250°, 4 mph	Method Blank	1.42
		Field Blank	1.64
		Site 1	0.48
		Site 2	0.81
		Site 3	0.76
		Site 4	1.46
		Site 4 Duplicate	0.75

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APPENDIX A

DETAILED DESCRIPTION OF CONDUCT OF STUDY
MICHIGAN DIOXIN STUDIES
DOW CHEMICAL BUILDING 703 INCINERATOR EMISSIONS STUDY

APPENDIX A

I. SAMPLING METHODS

The following sections concern the selection of methods employed to detect the compounds of interest from the various media that were sampled. Reference is made to Tables V-1 and V-2 of this report, where the compounds are differentiated according to the analytical procedures necessary to detect them.

A. Precombustion Air

A high-volume air sampler modified for the collection of PCDD/PCDF, and another similar sampler for semi-volatile organic compounds, were placed at ground level between two and four meters from the rotary kiln combustion air intake. Each sampler consisted of a glass fiber filter of the type commonly employed in ambient air monitoring for particulate matter, followed by a cylindrical trap containing 25 grams of 16/50 mesh Amberlite XAD-2 resin, configured in a manner based upon that developed by Lewis et.al.[12,13]

Design flow rates for the two samplers were derived on the basis of calculated resin breakthrough volumes for the compounds of interest. For PCDD/PCDF, it was determined that a sampling flow rate of 1.1 to 1.5 m^3/min, and a total sample volume no greater than 720 scm, would be appropriate. For the other semi-volatile (semi-VOA) compounds a flow rate of 0.6 to 0.8 m^3/min was selected, to result in a final sample volume not to exceed 350 scm. In actual practice, however, both samplers operated at flow rates of approximately 0.7 m^3/min owing to the air flow resistance presented by the tightly-packed XAD-2 resin columns.

Volatile compounds (for VOA, or volatile organics analysis) were monitored utilizing a low-volume sampler patterned after that described by Riggin.[6] Sampling cartridges containing 1.5 grams of Tenax® GC [poly (2,6-diphenyl phenylene oxide)] were suspended approximately two meters above ground and three to four meters from the rotary kiln air intake. Air flow rates of 25 to 35 cm^3/min were maintained for eight-hour sampling periods, with a target sampled gas volume of 14.4 standard liters.

Field blank samples were procured for each of the three samplers on every sampling day. In addition, a duplicate sample specific to each sampler was provided on one of the three sampling days.

B. Liquid Waste Feeds

It was known prior to the sampling effort that the sources and composition of waste delivered to the incinerator through each nozzle were likely to change every two to four hours on average. Also, because many of the liquid wastes were described by Dow personnel as containing more than 15 percent of single compounds, special handling and extraction procedures, involving intermediate preparation of extracts by an EPA contractor laboratory prior to analysis by a second contract laboratory, were required. These procedures are described fully in Appendix B to this report. As extracts for semi-VOA and VOA analysis were obtainable from the same samples utilizing these procedures, it was necessary only to obtain single representative samples of each distinct waste stream for these compound classes. For PCDD/PCDF, a second sample was required. In summary, each waste stream was to be represented by a time-composited sample for PCDD/PCDF, held in a 500-mL hexane-rinsed amber glass bottle, and a pair of hexane-rinsed 40-mL clear-glass VOA vials with Teflon septa, each containing composited aliquots of wastes. For VOA, care was taken to avoid agitation of sampled wastes and minimize possible losses of the volatile compounds to be analyzed. In any event, no sampling procedure for compositing VOA samples was available.

For samples to be representative over time, it was planned to obtain portions of liquid waste every half-hour, avoiding periods in which waste changes were occurring. Thus, for an eight-hour sampling period, up to 17 individual sets of grab samples were projected to be composited manually on an equal-volume basis. However, in some cases few samples were taken where particularly viscous or fuming wastes were handled.

Field blank samples were obtained on all three test days; a single field blank represented nozzles "BA" and "BB" as the nozzles were spaced closely together, while another field blank was taken for nozzle "C". Three field duplicate samples were drawn, all on the second test day, of two wastes at nozzle "BB" and a single waste at nozzle "C".

The following sections describe the ways in which the liquid waste sampling plan was altered at each nozzle.

1. Nozzle "BA"

On all three test days, the origin of the liquid wastes flowing through this nozzle remained constant throughout the test periods. However, Dow Chemical personnel indicated the waste originated from a chlorosilane manufacturing process at the adjacent Dow Corning Corporation plant, and was a fuming material which reacted violently with moisture in air. As the contents of the tank truck connected to nozzle "BA" were reported to be well-mixed and manual compositing would have presented a hazard to sampling personnel, it was decided to obtain a single grab sample for PCDD/PCDF, and a pair of VOA samples, midway through each test day.

2. Nozzle "BB"

During the sampling periods, two distinct wastes were fed through nozzle "BB" on the first and second sampling days, and a single waste was burned on the third day. Composites for PCDD/PCDF were manually formulated from the grab samples taken every half-hour. For semi-VOA and VOA, compositing was also performed on the first sampling day but was found to be laborious, with a high risk of spillage of liquids. Therefore, on the second and third days, PCDD/PCDF composites continued to be created, but to avoid the risks associated with compositing the lower-volume semi-VOA and VOA samples, it was decided that the grab sample (pair of VOA vials) taken midway in time through each run of waste would be chosen for analysis to represent that waste.

An indicated previously, field blank samples were obtained on all three days in the vicinity of nozzles "BA" and "BB", to apply to both nozzles. Cleaned 500-mL amber glass bottles and 40-mL clear glass VOA vials were filled with methanol for this purpose. Field duplicate samples were taken of the two wastes processed on the second sampling day.

3. Nozzle "C"

On all three sampling days, the wastes fed through nozzle "C" remained relatively constant throughout the sampling day, so that only a single set of samples was required to represent each day. For PCDD/PCDF, these samples were composited from grabs taken every half-hour on the first day and, to accommodate time constraints, every hour on the third sampling day. Semi-VOA and VOA waste samples were taken at times approximating the midpoint of these tests.

On the second sampling day, nozzle "C" waste was particularly viscous, making representative compositing infeasible. Thus, a single set of grab samples for PCDD/PCDF and semi-VOA/VOA was obtained at the start of the test run; a field duplicate sample consisted of a second complement of grabs taken at the same time. Field blank samples for all three days were made up of methanol-filled sample containers kept closed in the vicinity of the nozzle "C" sampling area for the duration of the test periods.

C. Low-BTU Liquid Waste

A spigot near waste nozzles "BA" and "BB" was drawn to obtain samples every half-hour for PCDD/PCDF and semi-VOA. Equal volumes of this liquid were taken and placed directly into composite bottles at these times. For volatile organic analyses (VOA), grab samples were obtained every half-hour; however, as no feasible method of compositing these samples was available, one sample taken midway through the sampling period was selected for analysis. Field blank samples, consisting of deionized water-filled sample containers, were taken on each day.

D. Incinerator Exhaust

1. Modified Method 5 (MM5) Trains for PCDD/PCDF and Semi-Volatiles

Two trains were operated simultaneously in sampling ports placed 90° apart in the exhaust duct downstream of the electrostatic precipitator. Each sample train, constructed as shown in Figure A-1, and based on previous designs of the MM5 train, consisted of a glass-lined, heated probe terminating in a stainless steel button-hook nozzle and attached thermocouple and pitot tubes. The probe outlet was attached to a glass filter holder containing a tared glass fiber filter (Reeve Angel 934 AH) maintained at a temperature of 248°F ± 25°F in an electrically-heated oven. Following the filter, sample gas passed through

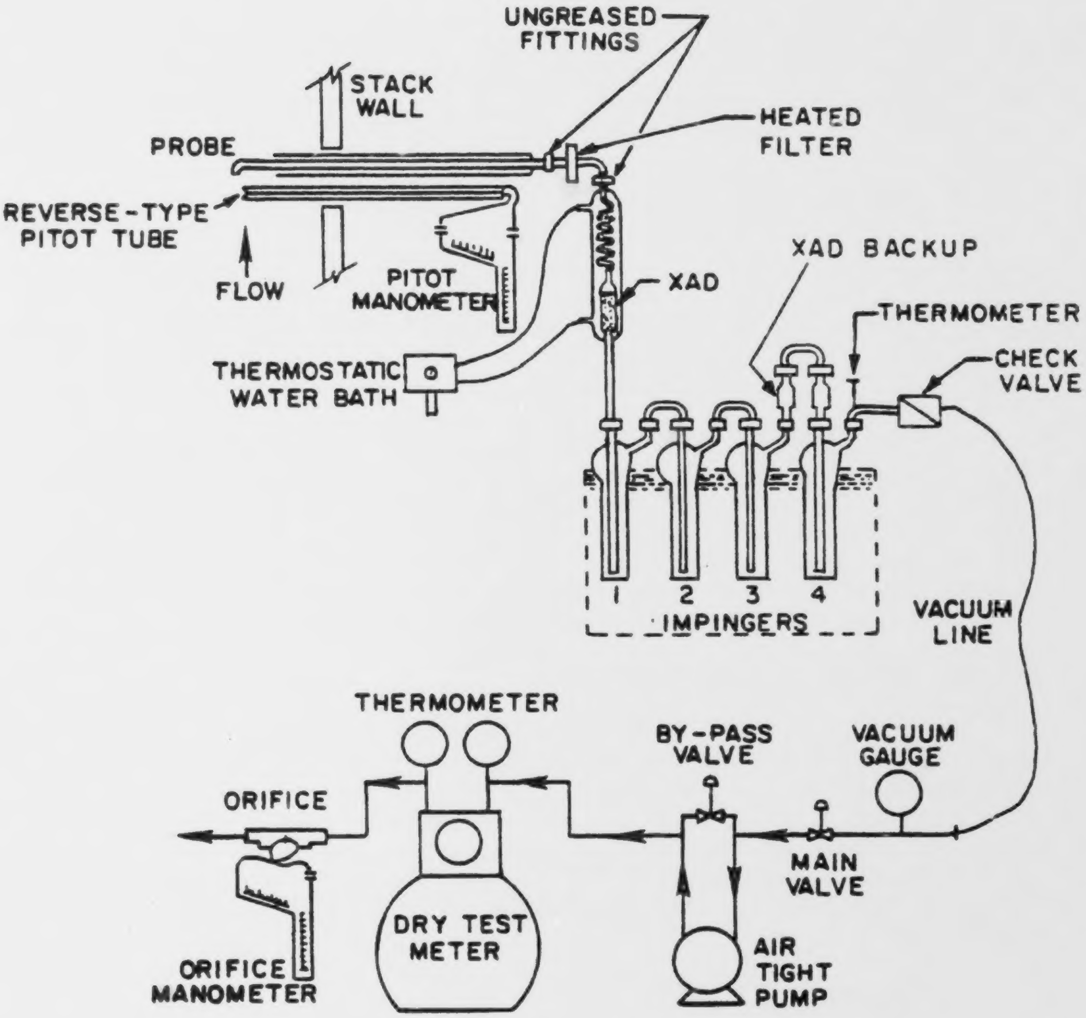

UNGREASED FITTINGS
STACK WALL
HEATED FILTER
PROBE
REVERSE-TYPE PITOT TUBE
FLOW
PITOT MANOMETER
XAD
XAD BACKUP
THERMOMETER
CHECK VALVE
THERMOSTATIC WATER BATH
1
2
3
4
IMPINGERS
VACUUM LINE
THERMOMETER
BY-PASS VALVE
VACUUM GAUGE
ORIFICE
MAIN VALVE
DRY TEST METER
AIR TIGHT PUMP
ORIFICE MANOMETER

FIGURE A-1

MODIFIED METHOD 5 EXHAUST GAS SAMPLING TRAIN flexible Teflon tubing to a water-cooled module containing approximately 25 grams of XAD-2 resin. A thermostatically-controlled water bath maintained the sorbent temperature at 70°F or below.

Water condensed from the gas stream passing through the XAD-2 module was retained in an impinger fitted with a short-stem inlet to avoid sample gas bubbling through collected condensate. The second and third impingers each held long-stem inlets; the second impinger was filled with 100 mL of deionized water at the start of sampling, while the third impinger was empty. A backup sorbent cartridge containing 7.5 grams of XAD-2 was placed between the third and fourth impinger. The fourth impinger held approximately 200 grams of indicating silica gel to remove traces of water from the sampled gas. All connections within the trains were composed of nonreactive materials such as glass or Teflon, and no sealant greases were employed. Sampled gas flowed through a check valve, tubing with a vacuum pump connected in parallel with a bypass valve, a dry gas meter, and an orifice and manometer for instantaneous flow rate measurement.

As indicated previously, two trains configured as above were operated simultaneously at a location in which two sampling ports were placed 90° apart. Initial plans called for a sampling period of eight hours, to obtain sufficient volumes of sample extracts for replicate analysis, sample splitting, and archiving. However, on the first sampling day, air flow through both trains could not be maintained for longer than approximately 6 1/2 hours. Apparently, the resistance to flow presented by the sorbents in the train and possibly collected moisture was too great to be overcome by the pump powering the sampling train. As a result of this experience, the planned sampling period was reduced to six hours on the second and third sampling days.

Owing to time delays, and the risk of causing leaks in the sampling trains by moving them, both trains remained on the same traverse in the exhaust duct during all three sampling periods. Thus, the trains sampled each point twice on the same traverse; the traverses were alternated such that one pair of diameters was employed on the first sampling day, and the other pair of diameters was used for the PCDD/PCDF and semi-volatile trains on the second and third days. This was done to avoid unnecessary movement of sampling trains in the limited space available on the sampling platform, and was not anticipated to have any significant effect on analytical results.

Two field blank trains were assembled for each sampling day and allowed to remain undisturbed near the MM5 sampling area. Sorbents and impinger contents of the sample and blank trains for PCDD/PCDF were removed from the trains by the analytical laboratory, with the exception of the sampling probe wash, which was conducted by the field contractor and placed in an amber glass bottle. The sample and blank trains for semi-volatile compounds were disassembled and rinsed by the field contractor, and placed in containers for shipment to the analytical laboratory. Field duplicate samples were not obtained as both sampling ports were utilized simultaneously.

2. Volatile Organic Sampling Train (VOST)

The VOST was constructed consistent with configurations developed by Midwest Research Institute, as shown in Figure A-2. The train was composed of a heated glass-lined probe with a plug of glass wool placed at the tip to remove particulate matter. A series of condensers and organic resin traps followed the probe; the first condenser cooled the sample gas stream to condense water vapor. Sampled gas and condensed water vapor then passed through a cartridge containing 1.5 grams of 60/80 mesh Tenax GC®. Condensate was collected in the

first impinger; the second condenser and a trap containing approximately 1 gram of Tenax and 1 gram of activated charcoal were positioned to retain compounds having low breakthrough volumes. A second impinger and a drying tube followed the second sorbent trap, for residual moisture removal.

Sample temperatures were monitored with thermocouples at the outlet of the probe and the inlet of the first Tenax cartridge. Gas temperatures within the probe were maintained above 130°C to avoid premature condensation of volatile compounds; through the resin traps, gases were cooled to 20°C or below.

All of the VOST sampling runs with the exception of two were conducted for 40 minutes at sample gas flow rates of 0.5 liter per minute, resulting in a total collected volume of 20 liters. For the remaining two runs, a sampling rate of 1 liter per minute was maintained for 20 minutes; one of these runs was that in which a field duplicate sample was taken.

Five or six VOST runs were completed on each sampling day. For each run, the two sorbent tubes were submitted for analysis as single samples. Between runs, the sorbent cartridges were changed; however, the condensate impingers remained in place for entire sampling days and thus represented a composite of all of the runs. The sorbent cartridges were transferred to containers packed with activated charcoal for shipment to the analytical laboratory, while the contents of the condensate impingers were placed in 40 mL VOA vials. Head spaces in these vials were eliminated by the addition of distilled, deionized water.

In addition to the single field duplicate sample noted above, field blanks of the VOST were taken on each sampling day. These unexposed sampling materials remained in the sampling area for complete days while all of the VOSTs for that day were utilized. The cartridges and condensate impingers were then handled in the same manner as regular samples.

3. Tedlar Bag Samples for Vinylidene Chloride

Samples were collected for approximately one hour utilizing an apparatus as shown in Figure A-3. The sampling assembly consisted of a cleaned, evacuated Tedlar bag placed inside a rigid container. Prior to sampling, each bag was purged with prepurified nitrogen. The Teflon sampling tubing was attached to the Tedlar bag container by a quick-disconnect coupling.

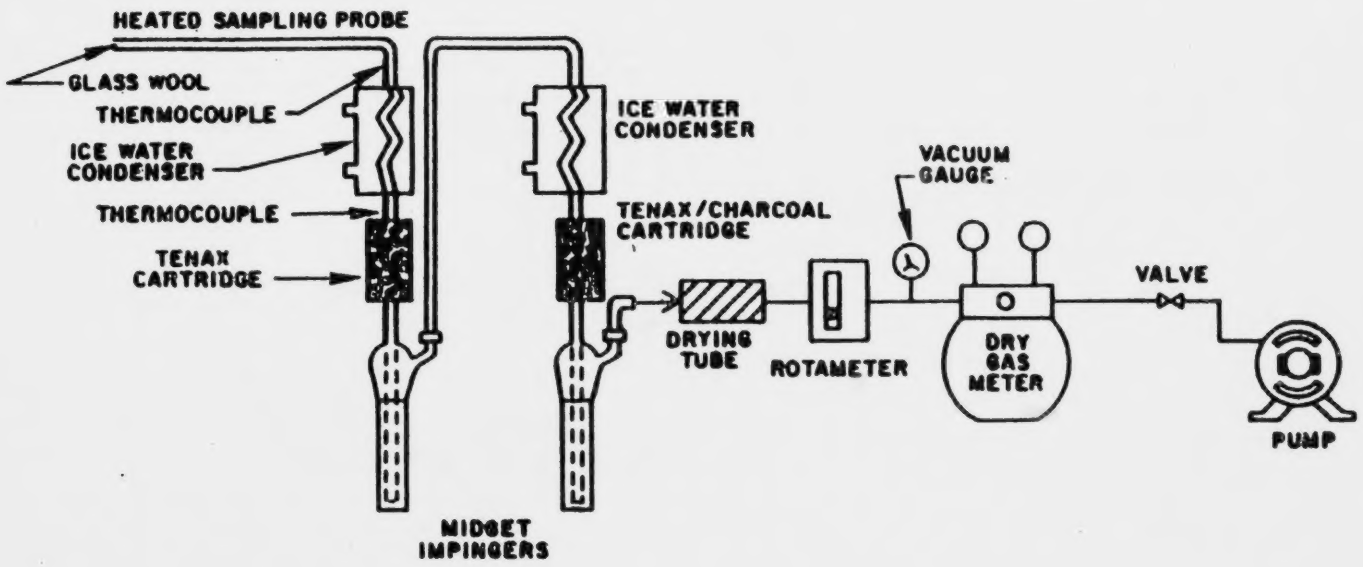
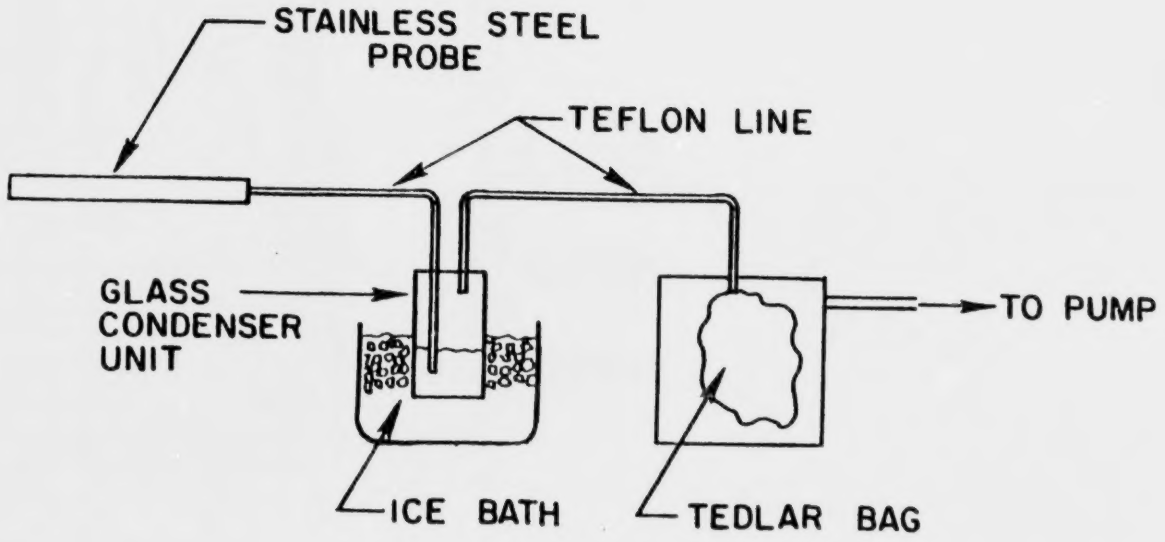

HEATED SAMPLING PROBE
GLASS WOOL
THERMOCOUPLE
ICE WATER CONDENSER
THERMOCOUPLE
TENAX CARTRIDGE
MIDGET IMPINGERS
ICE WATER CONDENSER
TENAX/CHARCOAL CARTRIDGE
DRYING TUBE
ROTAMETER
VACUUM GAUGE
DRY GAS METER
VALVE
PUMP

FIGURE A-2

VOLATILE ORGANIC SAMPLING TRAIN

FIGURE A-3

TEDLAR BAG SAMPLING SYSTEM FOR VINYLIDENE CHLORIDE


STAINLESS STEEL PROBE
TEFLON LINE
GLASS CONDENSER UNIT
TO PUMP
ICE BATH
TEDLAR BAG

Within two hours after sampling, filled bags were transported to a field laboratory in which direct analyses were performed with a gas chromatograph-electron capture detector (GC-ECD). One field bias blank, consisting of a bag filled with prepurified nitrogen, was analyzed daily. One collocated field duplicate sample was obtained on the second day of sampling. A description of the GC-ECD and its operating conditions are described in Table A-1.

4. Continuous Emissions Monitoring System

Incinerator combustion conditions were monitored utilizing a continuous emissions monitoring system (CEMS) assembled as shown in Figure A-4, consisting of a gas conditioning module, monitors for measurement of CO, CO_2, and O_2, and a data acquisition system. Samples were extracted from the exhaust gas stream at a point described previously; the effects of the carbon adsorption bed exhaust, described in Section V of this report, on the measured flue gas components were expected to be minor.

Sampled gas passed through a glass fiber filter for particulate removal, and then to a two-stage drier composed of a condenser and permeation drier. Conditioned gas was analyzed with the instruments detailed in Table A-2.

Exhaust gas was to be monitored for the duration of each Modified Method 5 test run. However, equipment startup problems, and the occasional necessity to utilize the sampling location for other measurements, prevented the continuous use of the CEMS. To supplement and check the CEMS, integrated samples were also obtained and analyzed using an Orsat analyzer.

E. Incinerator Ash

As indicated previously, samples of this material were taken from a dragout chain serving the ash trough. The chain was known from prior inspections of the facility to be started manually by an operator, approximately every hour on the hour. Therefore, a representative of the field contractor was present every hour to take, or supervise Dow personnel taking, portions of the solid material lifted out of the ash trough on an appropriate number of flights on the dragout chain. Typically, this meant samples were taken from three to five flights per hour; there was insufficient solid material remaining on the dragout chain to sample more flights than this. On occasion, fewer than three flights were sampled when ash removal was particularly light. Large pieces of incompletely-burned wood or fused metal were avoided in sampling owing to their unrepresentativeness when related to the full sample, and the impossibility of providing representative split samples to Dow Chemical and EPA contract laboratories.

Individual grab samples were taken from the chain utilizing a hexane-rinsed aluminum scoop mounted on a pole, and placed in a hexane-rinsed five-gallon glass jug to be held for later compositing and sample splitting. Compositing was performed by later emptying the jug contents on a floor or ground area which was covered first with a clean sheet of cardboard, in an area well separated from the incinerator, mixing and quartering them, and apportioning quarters with a cleaned scoop into separate washed glass containers for Dow and EPA analysis.

TABLE A-1

GC-ECD OPERATING CONDITIONS FOR VINYLIDENE CHLORIDE ANALYSIS DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

Instrument	Perkin Elmer 3920
GC Conditions	
Column	20% SP2100/0.1% carbowax 1500 on 100/120 supelcoport 10' x 1/8" SS Column
Temperature program	Isothermal at 50°C
Injector temperature	110°C
ECD temperature	325°C
Carrier flow	25 ml/min, argon/methane
Loop Conditions	
Volume delivered	1 ml
Loop temperature	125°C

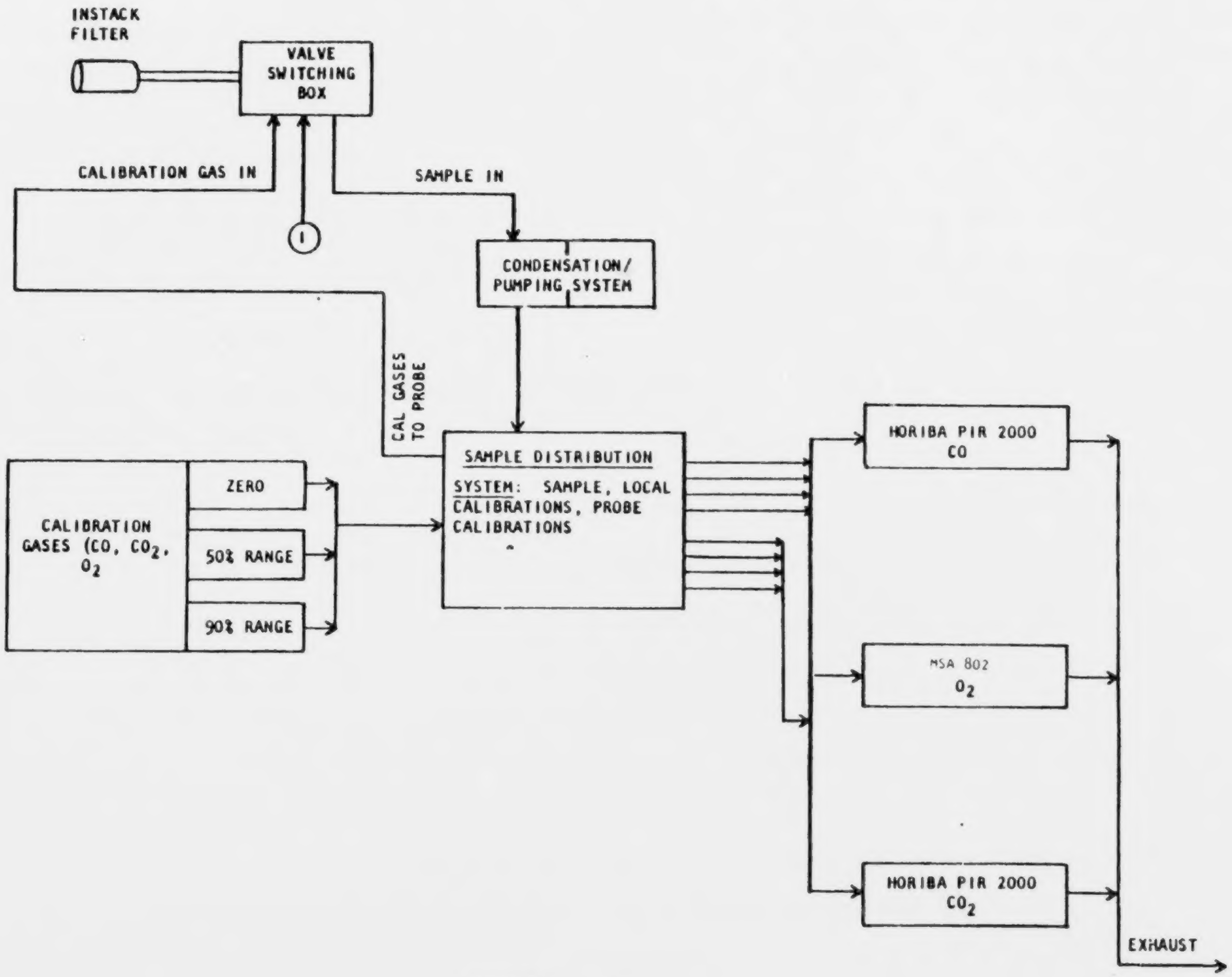

INSTACK FILTER
VALVE SWITCHING BOX
CALIBRATION GAS IN
SAMPLE IN
1
CONDENSATION/ PUMPING SYSTEM
CAL GASES TO PROBE
SAMPLE DISTRIBUTION SYSTEM: SAMPLE, LOCAL CALIBRATIONS, PROBE CALIBRATIONS
CALIBRATION GASES (CO, CO_2, O_2
ZERO
50% RANGE
90% RANGE
HORIBA PIR 2000 CO
MSA 802 O_2
HORIBA PIR 2000 CO_2
EXHAUST

FIGURE A-4

SCHEMATIC DIAGRAM
CONTINUOUS EMISSIONS MONITORING SYSTEM

TABLE A-2

CONTINUOUS EMISSIONS MONITORING SYSTEM OPERATING CONDITIONS

	Horiba PIR 2000 CO_2 analyzer	Horiba PIR 2000 CO analyzer	MSA O_2 analyzer
Operating sensitivity ranges	0-5% CO_2, FS 0-15% CO_2, FS 0-25% CO_2, FS	0-1000 ppm CO, FS 0-3000 ppm CO, FS 0-5000 ppm CO, FS	0-5% O_2, FS 0-10% O_2, FS 0-25% O_2, FS
Operating temperature ranges	24°F - 122°F	24°F - 122°F	32°F - 109°F
Analysis method	Nondispersive infrared	Nondispersive infrared	Paramagnetic wind
Linearity	± 1% FS	± 1% FS	± 1% of Full Scale
Accuracy	± 1% of Full Scale	± 1% of Full Scale	± 1% of Full Scale
Drift	± 1% of Full Scale in 24 hours in both zero and span	± 1% of Full Scale in 24 hours in both zero and span	<5% Full Scale for 24 hours in both zero and span
Noise level	<0.5% of Full Scale in most sensitive range	<0.5% of Full Scale in most sensitive range	<.25% of Full Scale in most sensitive range

Water entrained with sampled ash was allowed to drain, as much as possible, out of the samples. Ash trough water was sampled separately.

F. Influent and Effluent Water and Control Device Ash

1. Influent Service Water

Grab samples of influent service water (returned secondary treatment water) for PCDD/PCDF and semi-volatiles were taken taken every half-hour during the first and second sampling days, and, to accommodate time constraints, every hour on the third day. Individual samples were obtained in a washed and hexane-rinsed bottle, the contents of which were placed in a washed and hexane-rinsed brown glass one-gallon bottle for compositing on an equal-volume basis. For VOA, a pair of single grab samples was taken directly into washed 40-mL vials with Teflon septa, midway through each sampling period.

2. Effluent Waters

Effluents, as described previously, arose from the quench tower, venturi scrubber and demister (combined stream), electrostatic precipitator, and ash trough. Each of these streams was sampled utilizing ISCO automatic sampling devices, for PCDD/PCDF and semi-volatiles, and by taking single grab samples for VOA, as detailed above for influent service water.

The automatic samplers were set to draw a volume of water every half-hour during the incinerator exhaust sampling period, sufficient to fill to an appropriate level a five-gallon clear-glass bottle (washed with deionized water, methanol, and methylene chloride, and oven-dried) held inside it. The bottle was surrounded with ice for preservation of the sample. At the conclusion of sampling, portions of this total sample were poured into washed and hexane-rinsed one-quart brown-glass bottles to be submitted to the analytical laboratories for PCDD/PCDF and for semi-volatile compounds.

For VOA, each sampling location was represented by filling a single pair of 40-mL vials at a time corresponding closely to the midpoint of each sampling run. At all four locations, this process necessitated transferring samples from direct sampling containers, such as a large clear glass bottle, into the vials. In all cases, care was taken to fill the vials in a quiescent manner such that the head spaces were devoid of gases.

3. Control Device Ash

The Tier 4 Dioxin Strategy referenced previously required analyses of control device ash; the control devices at the Dow Chemical incinerator collected solid particles which were dispersed in water. Therefore, PCDD/PCDF and semi-volatile compounds in each of the four effluent water streams were analyzed separately in the aqueous and filterable solid phases. The latter analysis was estimated to be a reasonable representation of the presence of the analyzed compounds in the particulate or ash fraction of the control device water discharges.

II. SAMPLE IDENTIFICATION, HANDLING, AND CUSTODY

Samples were obtained by employees of the field contractor, GCA/Technology Division (GCA), and labeled according to a predetermined coding system. Where multiple grab samples were taken for compositing or transport out of the Dow Chemical facility, the samples were generally held in closed coolers near the individual sampling points; these coolers were inspected periodically to guard against tampering. Incinerator ash samples were stored in closed jars adjacent to the dragout chain in a location where visual custody was maintained by GCA or EPA personnel. Likewise, automatic samplers used for effluent water sampling were set in areas in which they were open to constant view.

As sampling was performed on one day and generally shipped to analytical laboratories on the next day, it was necessary to hold samples overnight prior to packing and logging. Two lockable trailers, one near the incinerator and the second placed on Dow Chemical property immediately outside the plant fence line, were used for secure storage.

Sample compositing and splitting were performed by or under the direct control of GCA personnel. After samples were placed into appropriate containers for shipment, they were relabeled to enable quick identification by contract analytical laboratories. A master cross-referenced list of samples and their identifying labels was formulated and maintained by the EPA project manager.

Sample containers were arranged as appropriate in shipping coolers and log sheets were completed to describe all of the samples in each cooler. On the first sampling day, the log sheets were written manually on standard EPA manifold custody forms; on the second and third sampling days, custody forms were created and reproduced using a computer and printer. Each individual cooler was packed with coolant and shock-absorbing material, and closed and sealed with custody tape imprinted with GCA identification. The samples were shipped to the analytical laboratories via Federal Express.

Information on liquid waste feedstocks was obtained from Dow Chemical prior to the start of sampling. Dow Chemical indicated that many or most of these wastes were composed of 15 percent or more of a single constituent. Therefore, liquid waste samples and blanks (made up of methanol) required special handling as "high-hazard" materials. These wastes were composited (where compositing was done) and placed into the smallest appropriate container, in this case 40-mL vials. Specialized tracking records were completed for each distinct sample, and all such samples were packed consistent with Department of Transportation regulations for flammable liquids or flammable-corrosive liquids, and shipped to an intermediary laboratory for extraction.

The above discussion applied to all samples with the exception of the Modified Method 5 PCDD/PCDF sampling trains, and the liquid waste samples analyzed for PCDD/PCDF. After sampling, these samples were stored in the contractor trailer outside Dow Chemical property until the conclusion of the three days of sampling; appropriately labeled, packed, and logged; and transported by automobile to the analytical laboratory.

III. ANALYTICAL PROCEDURES

Procedures for analyzing samples for semi-volatiles and volatiles are contained in Reference 7 to this report, while PCDD/PCDF methods are indicated in References 7 and 8. For convenience, the specific analytical procedures and quality assurance aspects relating to analyses of PCDD/PCDF by the contract laboratory, the Brehm Laboratory, Wright State University, are excerpted from Reference 8 and presented as Appendix C to this report.

A. Semi-Volatiles and Volatiles

Volatile pollutants, generally those with boiling points lower than 100°C, were analyzed according to EPA Method 624. Water samples, including the incinerator influent and effluents, and VOST impinger liquids, were concentrated and analyzed directly using this method. However, solid sampling media (Tenax and charcoal) were desorbed in a Nutech thermal desorption unit at 190°C for 10 minutes at 30 mL/min with helium, directly onto the head of the GC column, which was held at 20°C.

Semi-volatile pollutants with boiling points above 100°C were analyzed using EPA Method 625 for base/neutrals and acids. As with volatile component water samples, impinger washes were concentrated and analyzed. In the Modified Method 5 train, front half samples (probe washes and filter) samples were analyzed as a unit. To accomplish this, the probe wash was concentrated and the filter extracted separately, and the fractions were combined before analysis. Results were typically reported in ug/L as the relative weight of probe wash was much greater than that of the filter. The filter, XAD-2 resin samples, samples of incinerator ash, and the solid filtrates from effluent waters were Soxhlet extracted with methylene chloride for 16 hours in preparation for analysis. All analyses were performed in a Finnigan model 4000 GC/MS.

B. PCDD/PCDF

As indicated above, References 7 and 8, and Appendix C to this report contain descriptions of the methods used to analyze samples for PCDD/PCDF, and specific TCDD isomers.

C. Tedlar Bag Samples for Vinylidene Chloride

Whole-air samples were analyzed on a Perkin Elmer model 3920 GC/ECD maintained under the conditions shown in Table A-1. The gas chromatograph was calibrated prior to each daily run with zero gas and four typical upscale vinylidene chloride concentrations: 27, 50, 111, and 235 ppb. A fifth upscale concentration, 531 ppb, was added when measured vinylidene chloride concentrations exceeded 235 ppb.

Gas samples were taken for periods of 30 to 65 minutes, such that bags were filled with a volume sufficient to be analyzed. As each sample was analyzed in triplicate, the analytical process typically required a longer time than did sample collection, prompting concerns about the stability of samples while being held for analysis. Therefore, three bag samples were reanalyzed on the day following the first and third sampling days. The results of these tests indicated good sample stability over nearly 24 hours' holding time, and suggested that reactions, leaks, or other changes occurring in samples being held for one to four hours before analysis were not significant. Sample bags were used only once and then discarded, to avoid contamination or wall effects from sample to sample.

D. Continuous Emissions Monitoring System (CEMS)

The arrangement of the continuous emissions monitoring system employed to analyze incinerator exhaust gases has been described previously. The specifications (see Table A-2) of the system show goals for relative accuracy and zero and span drift. Results of Orsat analyses for oxygen and carbon dioxide were compared with average data from the CEMS to derive relative accuracy comparisons; as carbon monoxide concentrations were below the range of sensitivity of the Orsat, it was not possible to evaluate relative accuracy with respect to CO. Zero and span drift were determined approximately six weeks after the completion of the study, and the results showed the following:

Instrument	Zero Drift (%)	Span Drift (%)
MSA 802 O_2 analyzer	0.00	0.52
Horiba PIR-2000 CO_2 analyzer	0.00	0.00
Horiba PIR-2000 CO analyzer	0.00	1.09.

These results compared favorably with the criteria shown in Table A-2.

IV. WASTES INCINERATED AND INCINERATOR OPERATIONS

Dow Chemical provided general information concerning the types of liquid and solid waste materials incinerated on each sampling day. In addition, basic descriptions of the chemical composition of each of these wastes were furnished, as every waste was labeled with a serial number corresponding to an analytical form filed internally by the company.

A. First Sampling Day - August 28, 1984

Company information indicated the wastes burned on this day consisted of bulk rubbish; drums and fiber packs (containerized solid wastes); and liquid wastes fed through all four input nozzles, including that for low-BTU liquid waste. These wastes are described below:

1. Rubbish

Bulk rubbish consisting of paper, cardboard, plastics, and wood was input continuously throughout the sampling period, at an average rate indicated by Dow Chemical to be 19.9 cubic yards per hour, or about 9950 pounds per hour.

2. Containerized Solid Wastes

A total of 84 containers of solid waste were incinerated between 1235 and 2000 EDT; below are general descriptions of each.

Dow ID Number	Number Fed	Total Weight (lbs)	Primary Constituents
1425-04	6	267	Glass, plastic filters
137-02	18	approx. 3000	Latex, plastic wastes, rubber
1244-01	5	381	Acrylamide, acrylonitrile
1202-03	1	120	Glass, toluene, ethanol
2603-01	13	approx. 600	Plastic and saran wastes
Q8-6039-01	8	1420	Filter aids, silicones, hydrocarbons
8793-01	21	2954	Miscellaneous Styron wastes
1552-02	10	1322	ABS resin
2603-02	1	90	Mineral spirits, methanol, MEK
2521-06	1	89	Glass, PVC, tars
(Total)	84	approx. 10200	

3. Nozzle "BA" Feed

A single waste, identified as number Q8-6011-01 and consisting of chlorosilanes, benzene, chlorobenzene, toluene, and other hydrocarbons, was fed from a tank truck. The Dow Corning facility located near the Dow Chemical plant was the source of the waste. The average flow rate of this waste was estimated by Dow Chemical as 900 pounds per hour.

4. Nozzle "BB" Feeds

Two liquid waste mixtures were fed. From 1235 until 1606 EDT, wastes from a storage tank were delivered at an average rate of 1764 pounds per hour. The components of this mixture were reported by Dow Chemical as follows:

Dow ID Number	Primary Constituents
8420-01	Sodium acetate, Dowanol, toluene
8440-03	Amines, Dowanols
8492-06	Polyoxyalkylene ether
8531-01	Alkanolamines, ethyl alcohol
8585-02	Butylene glycol, butylene oxide

From 1606 until the end of sampling at 2000 EDT, 972 pounds per hour of waste 1450-05 were fed from a direct-burn trailer. Dow's waste description showed this waste to be composed of 85% methanol and 15% ammonia.

5. Nozzle "C" Feed

Waste 1546-01 was delivered from a tank trailer at an average rate of 2360 pounds per hour. This waste was described by Dow as containing ethanol, toluene, acetone, and about 2% Probucol in water.

6. Low-BTU Liquid Waste

From 1400 until the end of sampling, approximately eight gallons per minute (4000 pounds per hour) of collected precipitation were fed to the incinerator.

7. Incinerator Operational Characteristics

No abnormal operating phenomena were cited by Dow personnel. A summary of incinerator operating data recorded at 15-minute intervals by Dow personnel throughout the sampling day appears in Table A-3, and in Table A-4 are exhaust gas oxygen, carbon dioxide, and carbon monoxide data as measured by the previously-described continuous emissions monitoring system; note that this system operated only during the second half of the first sampling day.

B. Second Sampling Day - August 30, 1984

Incinerated wastes included bulk rubbish; drums and fiber packs; and liquid wastes fed through all but the low-BTU liquid waste nozzle.

1. Rubbish

A continuous feed of loose solid waste was provided, at an average rate of 17.1 cubic yards per hour, or about 8550 pounds per hour.

2. Containerized Solid Wastes

Between 1005 and 1630 EDT, 73 containers described below were incinerated.

Table A-3

Incinerator Operational Data

	8/18/84 1235-2000	8/30/84 1005-1630	9/5/84 1010-1630
Rotary Kiln Temperature (°C)	823-1016	851-1089	877-998
Afterburner Temperature (°C)	1038-1106	1013-1096	1013-1121
Quench Water Flow (gpm)	703-717	706-724	719-727
Venturi Scrubber Water Flow (gpm)	265-276	252-264	207-223
Venturi Differential Pressure (in. H_2O)	26.3-28.7	20.7-25.8	16.6-19.4
Demister Water Flow (gpm)	961-989	961-985	968-987
ESP Water Flow (gpm)	169-177	172-176	160-181

Table A-4

Exhaust Gas Data
As Measured by Continuous Emissions Monitor

		Oxygen (%)		Carbon Dioxide (%)		Carbon Monoxide (ppm)	
Date	Time Measured (EDT)	Average*	Std. Deviation	Average*	Std. Deviation	Average*	Std. Deviation
8/28/84	1620-2030	11.76	0.35	6.73	0.47	47.5	16.7
8/30/84	1120-1650	12.74	0.34	6.00	0.49	62.7	55.9
9/5/84	1030-1710	11.28	0.82	6.21	0.50	32.4	22.7

* Arithmetic averages of ten-minute-averaged data during measurement period cited.

Dow ID Number	Number Fed	Total Weight (lbs)	Primary Constituents
1202-05	19	1159	Wood fiber
8793-01	4	647	Miscellaneous Styron wastes
8893-13	43	4292	Styrene, acrylonitrile, ethylbenzene
1245-05	3	128	Unspecified polymer
1136-01	4	24	Miscellaneous laboratory wastes
(Total)	73	6250	

3. Nozzle "BA" Feed

Approximately 1800 pounds per hour of Dow Chemical waste Q8-6011-01, the same as burned on the first sampling day, was fed to the rotary kiln through this nozzle.

4. Nozzle "BB" Feeds

From 1000 until 1415 EDT, wastes from a storage tank, consisting of a mixture of the following, were fired at a rate of 682 pounds per hour:

Dow ID Number	Primary Constituents
8420-01	Sodium acetate, Dowanol, toluene
8440-03	Amines, Dowanols
8440-05	Brake fluids, Dowanols, Dowfroth, polyglycols
8492-01	Acrylamide/acrylic acid copolymer
8492-06	Polyoxyalkylene ether
8531-01	Alkanolamines, ethyl alcohol
8585-02	Butylene glycol, butylene oxide
8769-01	Styrene, benzene, ethylbenzene wastes

From 1415 until the end of sampling, another tank mixture, described below, was fed to this nozzle at a rate of 1200 pounds per hour:

Dow ID Number	Primary Constituents
8052-04	Dimethyl sulfoxide, sodium chloride
8052-07	Dimethyl sulfoxide, dimethyl phthalate, tars.

5. Nozzle "C" Feed

Viscous liquids stored in a stationary tank were fed to the afterburner section of the incinerator at a rate of 1171 pounds per hour. The tank contents were a mixture of the following:

Dow ID Number	Primary Constituents
9018-03	#2 Diesel oil
9026-01	Phenolic tars, p-phenylphenol.

6. Low-BTU Liquid Waste

No wastes of this kind were incinerated on this date.

7. Incinerator Operational Characteristics

These data appear in Table A-3. Air pollution control equipment operations were normal, with the exception of a period from 1515 EDT until the end of sampling, in which occasional arcing was noted in the electrostatic precipitator, the result of water bridging between the emitting plate and the sidewall retaining bolts. Facility personnel indicated such arcing would ordinarily have triggered the shutdown of the incinerator to allow cleaning of the interior of the precipitator, had it become more severe.

In Table A-4, data concerning exhaust gas characteristics appear. Of particular interest are the relatively high CO concentrations measured. This reflects comparatively high peak CO values recorded at intervals corresponding to the introduction of containerized solid wastes to the incinerator, or approximately every six minutes. On several occasions, CO measurements exceeded the scale of the monitor (0 to 1000 ppm); as a result of these sharp peaks, the standard deviation of these measurements is also high.

C. Third Sampling Day - September 5, 1984

Incinerated wastes included bulk rubbish; drums and fiber packs; and liquid wastes from all four input nozzles during the sampling period, 1010 to 1630 EDT.

1. Rubbish

Loose rubbish was fed continuously at an average rate of 20.8 cubic yards per hour, or about 10,400 pounds per hour. Most of these wastes consisted of cardboard, wood, and plastic; a small portion was described as wet, and some scrap fiberglass insulation was incinerated.

2. Containerized Solid Wastes

A total of 58 containers of solid waste were incinerated at a uniform rate between 1010 and 1630 EDT. Their contents are described below:

Dow ID Number	Number Fed	Total Weight (lbs)	Primary Constituents
358-07	1	166	Demolition wastes
1586-07	17	812	Dowco 453ME
1250-02	2	143	Miscellaneous laboratory wastes
1223-01	2	approx. 250	Miscellaneous laboratory wastes
1156-01	1	111	Miscellaneous waste solvents
1145-01	1	174	Organic solvents
1224-08	8	409	DMSO, perchloroethylene
1224-02	1	177	Miscellaneous laboratory wastes

1407-07	10	1459	Polyethyloxazoline
1215-04	1	57	ABS, ethylbenzene, styrene
1215-02	1	81	Styrene, ethylbenzene
8428-03	12	3375	Sodium trichloropyridinate
1584-02	1	106	Dursban, methylene chloride in solid sorbent
(Total)	58	7320	

3. Nozzle "BA" Feed

Dow Corning wastes were incinerated at an average rate of approximately 1726 pounds per hour. As indicated previously, this waste, number Q8-6011-01, was composed of chlorosilane, benzene, chlorobenzene, toluene, and other hydrocarbons.

4. Nozzle "BB" Feeds

A mixture of the following liquid wastes was incinerated at an average rate of 3002 pounds per hour.

Dow ID Number	Primary Constituents
688-03	Waste oils, chloroethylene, ethylene glycol
8020-01	Methyldiethanolamine
8420-01	Sodium acetate, Dowanol, toluene
8440-03	Amines, Dowanols
8440-05	Brake fluids, Dowanols, Dowfroth
8492-01	Acrylamide/acrylic acid copolymer
8492-06	Polyoxyalkylene ether
8531-01	Alkanolamines, ethyl alcohol
8585-02	Butylene glycol, butylene oxide
8769-01	Styrene, benzene, ethylbenzene wastes

5. Nozzle "C" Feed

A mixture of wastes referred to as "Canada-02" was delivered from a tank truck at a rate of 1758 pounds per hour. Chemical composition data provided by Dow Chemical indicates the waste consisted primarily of styrene, with the following constituents also present, in descending order: carbon tetrachloride, 4-vinyl cyclohexene, benzene/butadiene, ethylbenzene, isopropyl benzene, and n-propylbenzene.

6. Low-BTU Liquid Waste

A mixture of aqueous wastes described by Dow Chemical as collected precipitation, condensate from tank storage area carbon bed regeneration, and water from hydroblasting cleanup, was fed to this nozzle at a steady rate of 4754 pounds per hour between 1130 and 1630. Before this, water flow was intermittent.

7. Incinerator Operational Characteristics

No operational abnormalities were reported by Dow personnel. Tables A-3 and A-4 contain operational data and exhaust gas measurements obtained through continuous emissions monitoring.

APPENDIX B

EXTRACTION PROCEDURE FOR "HIGH-HAZARD" LIQUID WASTE SAMPLES

FRED C. HART ASSOCIATES, INC.

MICHIGAN DIOXIN STUDIES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR EMISSIONS STUDY

Method: RSL-901
Page: 1 of 5
Date: June 1984
Replaces: All previous editions

Separation and Aliquoting High Hazard Waste Samples

1. Scope and Application

This is a general purpose method that provides procedures for phase separating and aliquoting high hazard waste samples taken from drums, lagoons, tanks, landfills, and other uncontrolled hazardous wastes. The method is applicable to a wide range of analyses including volatile organics, semi-volatile organics, total metals, spot tests, and strong acid anions.

2. Summary of Method

2.1 Individual phases are separated by decanting and centrifuging. After separation, phases are weighed to a tenth of a gram and recomposited by percent weight (except for compositional analysis). Prior to recomposition, liquid phases are tested for water miscibility.

2.2 Phase separation and recomposition is performed in order to obtain representative aliquots from the original sample.

3. Definitions

The characteristics of the samples defined below are the only descriptions to be used in describing the physical attributes of the sample:

Phase - A solid (gel or paste), water miscible liquid, non-water miscible liquid.

Paste - Inseparable solid and liquid.

Viscosity - Non-viscous, similar to water, or viscous.

Color - Colorless, light of the color, medium of the color, or dark of the color. Use only primary and secondary colors.

Texture - Fine grain (powdery), medium grain (sand), or course grain (large crystals).

Turbidity - Clear, cloudy (transmits light), or opaque.

Minor phase - Phases that represent less than or equal to 5% by weight for mercury aliquoting. Phases that represent less than or equal to 2% by weight for all other aliquoting.

4. Artifacts

Artifacts may occur in samples depending on the nature of the waste and how it is obtained. Artifacts are not minor phases but are due to extraneous agents not of the waste. When excluding a portion of a sample from recompositing based on the apparent presence of an artifact, the decision should be fully documented on the laboratory bench sheet.

5. Safety

High hazard samples are expected to contain concentrations of substances of unknown toxicity and carcinogencity up to 100% by weight. Thus, each sample is to be treated as a potential health hazard and exposure to these samples is to be minimized. Each analyst is responsible for maintaining awareness of safe handling procedures used in this method. The samples are collected, packaged, and shipped according to recommended procedures for hazardous wastes and are to be prepared using the following method in a Regulated Substances Laboratory prior to analysis.

6. Apparatus and Equipment

6.1 Radiation meter with pancake probe

6.2 Centrifuge, explosion-proof

6.2.1 large process type for 8 oz. jars

6.2.2 small type for vials

6.3 Vials and Jars

6.3.1 2 dram

6.3.2 40 mL

6.3.3 20 mL

6.3.4 8 oz. jar

6.3.5 4 oz. jar

6.4 Pipets, various sizes

6.5 Balance, four place

6.6 Spatulas, various types

6.7 Miscellaneous

6.7.1 Kimwipes

6.7.2 Soap and water squirt bottles

6.7.3 Methanol squirt bottles

6.7.4 Plastic bags, various sizes

6.7.5 Stainless steel trays

6.7.6 Teflon liners, various sizes

7. Sample Handling

Samples are removed from shipping cans inside a hood and repackaged after phase separation and aliquoting in the same manner. Only dilutions, digestions or extractions of a sample may be removed from the RSL; however, upon special request small amounts of undiluted samples may be taken from the regulated area.

8. Procedure

8.1 Traffic Report/Sample Verification

8.1.1 Verify Traffic Report against sample identification tag. If custody seal is present, sign and date where provided. Verify the information on the sample tag with the phase separation record. If there are any discrepancies, the sample tag is checked against the Chain-of-Custody record. The differences are recorded under sample tag information. Reconciliation is made bv Sample Control if necessary.

8.2 Place sample can inside small plastic bag. Remove lid from can and perform radioactivity check. If positive, replace can lid, remove gloves and vacate lab. Remove sample from can and record sample condition on Phase Separation Record and Traffic Report. Wipe down sample container with a Kimwipe moistened with soapy water.

8.3 Open sample container and again perform radioactivity check and record results. If positive, replace jar lid, remove gloves and vacate the lab area.

8.4 Complete any other header information on the phase separation record.

8.5 Phase Separation

8.5.1 If sample is a single phase liquid, test for water miscibility by adding several drops of sample into a 2 dram vial containing 0.5 mL of deionized water. Record results. Transfer 35 mL of the liquid to a labeled 40 mL vial or 2 oz. bottle. Recap original sample.

8.5.2 If sample is a single phase solid, transfer approximately 35g into a labeled 40 mL vial.

8.5.3 If sample is multi-phase, split sample into 2 jars, place the jars in plastic bags and centrifuge at 3000 rpm (50%). Centrifuge sample for not less than five minutes but no longer than ten minutes. Check for separation completeness. If incomplete, centrifuge for an additional five minutes.

8.5.4 Transfer each individual phase to appropriate tared and labeled vials or jars and record final weights on separation record. Perform water miscibility test as described in Section 8.5.1 on each liquid phase.

8.6 Describe and record each phase using phase descriptions in Definitions (Section 3).

8.7 Remove any material from outside of vials and jars with Kimwipes and soap and water. (Solvents may be necessary but use only on SEALED containers). Place contained phases in one plastic bag and store for future aliquoting.

8.8 Aliquoting

8.8.1 Ascertain whether aliquoting is for compositional or general charactertization analysis. For compositional analysis weigh a predetermined amount of phase into an appropriate test vial. For general characterization analysis, recomposite each phase by percent weight into an appropriate test vial. Refer to extraction and analysis methods for proper aliquot weights.

8.8.2 Unless requested, minor phases are not aliquoted. Minor phases are defined in Section 3.

9. Waste Disposal

9.1 All items listed in the following table will be placed in the appropriate waste container. The containers will be either labeled with the DOT classification from the table or be placed in another container which will be labeled with the DOT classification (e.g. plastic bags will be placed in a labeled 55 gallon drum).

Item	Container	Classification (DOT)
Waste Glass	5 Gallon can Reinke	Waste Flammable Solid
Waste Solvents	Waste Solvent Can	Waste Flammable Liquid
Waste Wood	5 Gallon can Reinke	Waste Flammable Solid
Waste Paper Gloves, etc.	Plastic Bag	Waste Flammable Solid
Waste Liquids Soapy H_2O, DDI	Waste Solvent Can	Waste Flammable Liquid

Approved by ______________________ Date ________________

Reviewed by ______________________ Date ________________

Method: RSL-902
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Date: June 1984
Replaces: All previous editions

Organic Chemical Extraction and Gas Chromatographic Screening of High Hazard Waste Samples

1. SCOPE AND APPLICATION

This is a general purpose method that provides procedures for preparation and screening of organic extracts for volatile organic (VOA), base/neutral/acid (B/N/A), and pesticide/PCB. High hazard waste samples include all chemical wastes both in containers, such as drums or tanks, and uncontained such as in piles, solid chemical or pooled liquids.

The method is directed to highly contaminated soil samples and waste samples that may be solid, aqueous liquid, or nonaqueous liquid and suspected to contain greater than 0.01% of any one organic chemical component. The method is not designed for waste samples expected to contain less than 10 ppm of base/neutral and acid priority pollutants; for example, as in many sediment samples taken from leachate streams. That type of sample should be analyzed using more traditional methods, such as Soxhlet extraction or homogenization, with larger sediment/soil samples.

2. SUMMARY OF METHOD

2.1 One to 1.5 gram aliquots of soil, solid, aqueous liquid, or nonaqueous liquid are transferred to vials and diluted with either methanol, hexane, or methylene chloride. Solid phase aliquots which are not soluble in the extracting solvent are sonicated for two minutes. All other aliquots are either shaken by hand or a mechanical wrist shaker for one minute.

3. DEFINITIONS

B/N/A - Base/Neutral/Acid

VOA - Volatile Organic analysis

External standard - a known amount of a pure compound that is analyzed with the same procedures and conditions that are used to analyze samples containing that compound. From measured detector responses to known amounts of the external standard, a concentration of that same compound can be calculated from measured detector response to that compound in a sample analyzed with the same procedures.

Internal standard - a pure compound added to a sample in known amounts and used to calibrate concentration measurements of other compounds that are sample components. The internal standard must be a compound that is not a sample component.

NEIC dirt - a loamy soil obtained near the NEIC/Denver which has been dried, crushed, and sieved in a #10 sieve.

Laboratory control standard - a solution of analytes prepared in the laboratory by dissolving known amounts of pure compounds in a known amount of solvent. In this method, the laboratory control standard is prepared by adding appropriate volumes of the secondary dilution standard solution and the internal standard/surrogate compound spiking solution to a known soil/water/oil matrix.

Laboratory replicates - three aliquots of the same sample that are treated exactly the same throughout laboratory analytical procedures. Analysis of laboratory replicates indicate precision associated with laboratory procedures but not with sample collection, preservation or storage procedures.

Laboratory reagent blank - a portion of reagent solvent processed in the same manner as the sample.

Secondary dilution standard - a solution of analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and laboratory control standards.

Stock standard solution - a concentrated solution containing a certified standard that is a method analyte, or a concentrated solution of an analyte prepared in the laboratory with an assayed reference compound. Stock standard solutions are used to prepare secondary standard solutions.

Surrogate compound - a compound that is not expected to be found in the sample, is added to the original environmental sample to monitor performance, and is measured with the same procedures used to measure sample components.

4. LIMITATIONS

The procedure is designed to allow detection limits as low as 10 ppm for volatile organic priority pollutants. The procedure is designed to detect extracts at 100 ppm for base/neutral and acid priority pollutants, 10 ppm for TCDD and PCB's, and 10 ppm for chlorinated pesticides; lower limits of detection, tenfold below these values, can be achieved on relatively clean samples by concentrating the extracts to 1 mL. Some samples, however, may contain high concentrations of chemicals that interfere with the analysis of other components at lower levels; the detection limits in those cases may be significantly higher. These extraction and preparation procedures were developed for rapid and safe handling of high concentration chemical waste samples. The design of the method thus does not stress efficient recoveries or low limits of detection of all components. Rather, the procedures were designed to screen, at moderate recovery and sufficient sensitivity, a broad spectrum of organic chemicals. The results of the analyses thus may reflect only a minimum of the amount actually present in some samples.

5. SAFETY

Potentially carcinogenic, mutagenic, toxic, and other hazardous materials may be present in these waste samples at concentrations up to 100 per cent. This procedure is intended for use in a Regulated Substances Laboratory to minimize personnel exposure and other hazards relating to the handling of the samples. In particular, good laboratory practices should be used to minimize exposure and contamination throughout the preparation and analysis of these types of samples. Each person is responsible for maintaining awareness of safe handling procedures used in this method.

6. REAGENTS

6.1 Sodium sulfate (anhydrous). Granular, analytical reagent grade, pre-extracted with methylene chloride or muffled at 400°c. for 3 hours before use to remove interferences.

6.2 Methylene chloride. Pesticide residue analysis grade, or equivalent.

6.3 Hexane. Pesticide residue analysis grade, or equivalent.

6.4 Methanol. Pesticide residue analysis grade, free of purgeable organics. Check by adding 10 uL to 5 mL of organic free water, and analyzing by GC/MS using the purge and-trap technique or direct injection by GC/HECD.

7. APPARATUS AND EQUIPMENT

7.1 Glass scintillation vials, at least 20 mL, with screw cap and aluminum foil liner.

7.2 Wooden tongue depressors. Dispose of after using to transfer solid samples.

7.3 Balance capable of weighing 100 grams to the nearest 0.01 gram.

7.4 Vials and caps, 2 dram for GC autosampler.

7.5 Disposable pipets, 10 mL. Pasteur pipets.

7.6 Gas chromatograph with a flame ionization detector and electron capture detector.

7.7 Ultrasonic probe, Braun-Sonic 1510 with intermediate probe attachment, or equivalent.

7.8 Test tube rack.

7.9 Glass vials with Teflon-lined screw caps, 12 mL for shipment of extracts.

7.10 VOA bottles, 20 or 40 mL with Teflon-backed septum and screw cap, for extraction and shipment of VOA samples.

7.11 Hamilton 10 ul and 250 ul gas tight syringes.

7.12 Glass wool rinsed with methylene chloride.

8. CALIBRATION

8.1 BASE/NEUTRAL/ACID ANALYSIS

8.1.1 Prepare stock external standard solution by weighing about 0.025 grams of pure phenanthrene-d10. Dissolve the material in methylene chloride, dilute to volume in a 20 mL volumetric flask. Dilute a portion of the stock solution (secondary dilution standard) to achieve a concentration of 25 ug/mL. Prepare stock internal standard solution by weighing about 0.050 grams of pure napthalene--d8 and phenanthrene-d10. Dissolve the material in methylene chloride, dilute to volume in a 10 mL volumetric flask. Transfer the stock standard solutions into Teflon sealed screw-cap bottles. Store at 4° C. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.1.2 Using an injection of 2 uL of the external standard solution, standardize the flame ionization detector for half-scale response.

8.1.3 Recommended operating conditions for the gas chromatograph are:
Thirty (30) meter X 0.25 mm bonded-phase silicone-coated fused silica capillary column with helium carrier gas at a flow rate of 30 cm/second. Column temperature programmed: isothermal, 50° C. for four minutes, then programmed at 8° C/minute to 300° C. Hold time, 15 minutes.

8.1.4 Concentrate 10.0 mL of the B/N/A Control and Reagent Blank extracts under a gentle stream of purified nitrogen to 1.0 mL.

8.1.5 Transfer the 1.0 mL extract to a 2 dram vial and seal.

8.1.6 Immediately prior to analysis, add 10 uL of the internal standard solution to the extract. The final concentration of the internal standards in the extract should be 50 ug/mL.

8.1.7 Surrogate compounds shall be quantified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given surrogate.

$$\frac{AI_x \quad AC_s}{AI_s \quad AC_x} \times C_x$$

AI_x = Area of Internal standard in standard
AI_s = Area of internal standard in sample
AC_s = Area of surrogate in sample
AC_x = Area of surrogate in standard
C_x = Concentration of surrogate in standard

8.1.8 Each chromatogram shall be clearly identified with the following information.

(a) Case or Project Number
(b) Sample Identification
(c) Fraction (BNA, VOA, Pesticide/PCB)
(d) Standard, Reagent Blank, Control
(e) GC run number
(f) If sample is a reagent blank or control, list GC number of Standard used for quantitation
(g) Date of analysis
(h) Analyst name
(i) Standard Operating Procedure number
(j) Each internal standard and surrogate identified.

8.1.9 Report results on QC Bench Sheet.

8.2 PESTICIDE/PCB PREPARATION

8.2.1 Prepare stock solution by diluting 1.0 mL of concentrated Aroclor 1254 (5000 ug/mL) to 10 mL in acetone. Final concentration to be 0.5 mg/mL.

8.2.2 Transfer the stock solution into Teflon-sealed screw-cap bottles. Store at $4^{O}c$. Stock standards should be checked frequently for signs of degradation or evaporation especially just prior to preparing calibration standards from them.

8.2.3 Using an injection of 2 uL of the secondary dilution standard, standardize the electron capture detector for half-scale response. The secondary standard is a 10x dilution of the stock solution.

8.2.4 Recommended operating conditions for the gas chromatograph are:

Supelcoport (100/120 mesh) coated with 1.5% SP-2250/1.95% SP-2401 packed in a 1.8 m long X 4 mm ID glass column with nitrogen carrier at a flow rate of 40 mL/minute. Column temperature, isothermal at 200°c.

8.2.5 Dilute the Pesticide/PCB control and Reagent Blank extracts by adding 100 uL of extract to 0.9 mL of hexane.

8.2.6 Surrogate compounds shall be quantified by the external standard method. The integrated area or peak height for the five largest and most resolved peaks are averaged:

$$\frac{A_s}{A_x} \times C_x$$

A_s = Average area of peaks in sample
A_x = Average area of peaks in standard
C_x = Concentration of surrogate in standard

8.2.7 Reporting (see paragraph 8.1.8)

9. QUALITY CONTROL

9.1 Two reagent blanks for each fraction (VOA, Pesticide/PCB, B/N/A) shall be prepared with each project or for every 20 samples within a project. One is analyzed at the RSL while the other is shipped with the sample extracts to the analysis laboratory.

9.2 One sample from each project or for every 20 samples within a project is prepared for spiking purposes by aliquoting six (extra) additional fractions. Three fractions are spiked at 50 ug/g of sample with PCB stock solution (Aroclor 1254), three more fractions are spiked at 100 ug/g of sample with Base, Neutral and Acid standards (See Table 1.)

9.3 Each B/N/A fraction, blank, and replicate spike shall be spiked with 150 uL of surrogate Spike. (see Table 1).

9.4 With each project or 20 samples within a project, the RSL will prepare two 1.5 gram multi-phase control samples by mixing 1.0 gram of NEIC "dirt", 0.1 gram of vegetable oil, and 0.4 gram of tap water. One control is spike with 150 uL of B/N/A surrogate mix, the second with 150 uL of PCB mix. The normal extraction procedure is followed. (See Table 1 for concentrations of these spike mixes.)

10. PREPARATION PROCEDURE

10.1 Transfer 1.5 + .04 g aliquots (1.0 + .04 g for VOA) to appropriate test vials (Method RSL-901, Section 8.8)

10.2 Dilute the VOA sample with 10 mL interference-free methanol. Disrupt insoluble solid samples by ultrasonic probe for 2 minutes at 100 watts power.

Cap, and shake all other samples for one minute. Note: vials should be capped and removed from the hood prior to working with methylene chloride or any other solvent in the hood. They should also be stored in a solvent-free atmosphere at $4^{o}c$.

10.3 Add 150 uL of B/N/A surrogate mix to each of the sample portions to be extracted with methylene chloride. Add the surrogate so that it is distributed as uniformly as possible over the sample; shake the sample to achieve better mixing if appropriate. In addition dilute 100 uL of B/N/A surrogate mix up to 10 mL in methylene chloride. This is to be used as the gc calibration standard for analyzing blanks and controls.

10.4 Add 15 mL of hexane to the pesticide/PCB fractions and 15 mL of methylene chloride to the B/N/A fractions. If the pH of the aliquot is less than or equal to five, or greater than or equal to eight, an additional B/N/A extract is prepared with pH adjustment. The pH adjustment is prepared by adding the equivalent amount of acid or base necessary to reach the end point of the acidity/alkalinity determination. Add 6N HCl to aliquots whose pH is greater than or equal to 8. Add 6N NaOH to aliquots whose pH is less than or equal to five. The pH adjusted B/N/A aliquot is not prepared when the addition of acid or base exceeds 2.0 mL.

Calculations for determining required acid or base additions.

$$\text{vol. of acid or base required for adj., mL} = \frac{1.5 \times A \times N_1 \times V_1}{B \times N_2}$$

A = dilution volume, mL
B = volume of aliquot, mL
N_1 = normality of titrant
N_2 = 6 (normality of adjusting soln.)
V_1 = volume of titrant required, mL

10.5 Add approximately 2.5 g of anhydrous sodium sulfate to each of the B/N/A and pesticide/PCB extracts to absorb any water. Additional sodium sulfate may be required.

10.6 Disrupt insoluble solid samples for 2 minutes using an ultrasonic probe at 100 watts power. Cap and shake all other samples for one minute.

10.7 Using a disposable 10 mL pipette, transfer 10 mL of the extract to a shipping vial. If the sample contains suspended solids that will not pass through glass wool, filter enough extract through a pasteur pipet loosely packed with 2-3 cm of glass wool to yield 10 mL of filtrate.

10.8 If a pH adjustment extraction was performed, add 5.0 mL of each methylene chloride extract together in a shipping vial; the final volume being 10 mL.

10.9 For all extract vials that are to be shipped, mark the liquid level on the side of the vial.

11. METHOD PERFORMANCE

11.1 The results of recovery studies presented in Section 11 are from the extraction of 1.0 gram samples with 10 mL of solvent. It should be noted that during sample extraction preparation, sodium sulfate is added to the sample prior to the sonication step rather than after sonication. This change raised recovery of the 50 ug/g PCB spike into the multi-phase control sample from 50-60 percent to 80-90 percent; recoveries of the B/N/A surrogate compounds were not detectably affected by the change. The data in Tables 2 through 6 show variability of recovery due to matrix, pH, solvent, concentration, and analyst. The B/N/A extracts for these studies were analyzed on an SE54 capillary column with an FID detector. The data in Tables 7 through 9 were obtained from capillary column GC/Ms analysis. The GC/MS analysis differed from that used by contractor laboratories in that only phenanthrene-d10 was used as an internal standard for quantitation, and a 15M DB5 column with a u um film thickness was used rather than a 30M 0.25 um film thickness column. Section 11.3 presents data showing the performance of the method for VOA compounds; losses of very volatile compounds (gases) on the order of 20-40 percent can be expected.

11.2 The data in Tables 7 through 10 were obtained from analysis of quadruplicate spikes into three matrices. Matrices 1 and 3 were real samples whose only criterion for selection for spiking was that the level of chromatographable organics would allow the final extract to be concentrated to 1 mL. Matrix 2 is the material referred to as "NEIC dirt" which is described in Section 3.

11.3 A possibility in the use of any extraction method for VOA compounds is the loss of volatile compounds during the extraction. In order to investigate the possibility of losses during the sonication step of this procedure, replicate portions of standards in methanol were sonicated for various lenghts of time. The results indicate that losses between 20 to 50 percent can be expected, using this extraction procedure for compounds which are gases at room temperature (chloroethane, methyl bromide, methyl chloride, vinyl chloride). Losses of other compounds ranged from negligible up to the order of ten percent for a sonication of two minutes. The developers of this method suggest that for the assumed application of this method, losses of ten percent can be considered negligible. Table 11 presents the results of the percent recovery as a function of sonication time study. Table 11 lists the average percent recovery and standard deviation for three determinations at each time. The sonication study involved six replicate portions of a standard solution in methanol. Three 10-mL portions were sonicated for one minute and 1 mL aliquots removed for analysis. The other three portions were sonicated for two minutes before removing aliquots. Each group of three aliquots for analysis after two and four minutes. This procedure gave aliquots for analysis after sonication times of one through six minutes. However, the sonication time for periods greater than two minutes is not continuous. Solutions had an opportunity to cool before the next two-minute sonication period; sonicating continuously for the time periods shown could be expected to produce lower recoveries because of increased heating of the solutions. The sonic probe was operated for sufficient time to bring the tip to a typical operating temperature before sonicating any of the VOA standards. The analyses were performed by GC/MS.

Approved by ______________________ Date ________________

Reviewed by ______________________ Date ________________

APPENDIX C

ANALYTICAL PROCEDURES FOR PCDD/PCDF
BREHM LABORATORY - WRIGHT STATE UNIVERSITY

MICHIGAN DIOXIN STUDIES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR EMISSIONS STUDY

7.0 GENERALIZED BREHM LABORATORY PROCEDURES FOR SAMPLE EXTRACT CLEAN-UP AND ANALYSES OF ENVIRONMENTAL SAMPLES FOR CDDs/CDFs

7.1 CLEAN-UP AND PRELIMINARY FRACTIONATION OF SAMPLE EXTRACTS

Extracts of the samples obtained as described in Section 5.0 are cleaned-up and fractionated using the following procedures.

7.1.1 Clean-up and Liquid Chromatographic Separation

a. Add 50 mL of doubly distilled water to the vessel containing the sample extract, reseal the vessel and agitate for 10 minutes. Allow the vessel to stand for a period sufficient for the aqueous and organic layers to separate completely, and remove and discard the aqueous layer.

b. Using the same procedure as applied in 3a., wash the extract successively with 50 mL portions of 50% KOH, doubly distilled water, concentrated H_2SO_4, and doubly distilled water, in each case discarding the washing agent. The acid washing procedure with concentrated sulfuric acid is repeated until the acid layer is visually colorless.

c. Add 5 g of anhydrous sodium sulfate to the washed extract and allow to stand in order to remove residual water. Transfer the extract to a centrifuge tube and concentrate to near dryness by placing the tube in a water bath at 55°C, and passing a gentle stream of filtered, prepurified N_2 over the solution.

d. Prepare a glass macro-column, 20 mm OD x 230 mm in length, tapered to 6 mm OD on one end. Pack the column with a plug of silanized glass wool, followed successively by 1.0 g silica, 2.0 g silica containing 33% (w/w) 1M NaOH, 1.0 g silica, 4.0 g silica containing 44% (w/w) concentrated H_2SO_4 and 2.0 g silica. Quantitatively transfer the concentrated extract from Step c. to the column and elute with 90 mL hexane. Collect the entire eluent and concentrate to a volume of 1-2 mL in a centrifuge tube, as before.

e. Construct a disposable liquid chromatography mini-column by cutting off a Pyrex 10 mL disposable pipette at the 4.0 mL mark and packing the lower portion of the tube with a small plug of silanized glass wool, followed by three grams of Woelm basic alumina, which has been previously activated for at least 16 hours at 600°C in a muffle furnace, and cooled in a dessicator for 30 minutes just prior to use. Quantitatively transfer the concentrate from Step d. onto the liquid chromatography column, rinse the centrifuge tube consecutively with two 1 mL portions of hexane, and also transfer the rinses to the chromatography column.

f. Elute the column with 15 mL of hexane and discard the eluent.

g. Elute the column with 10 mL of 8% (v/v) methylene chloride-in-hexane and discard the eluent.

h. Elute the column with 15 mL 50% (v/v) methylene chloride-in-hexane and retain the eluent. Concentrate just to dryness with a stream of nitrogen, as described above.

i. Take a 9-inch disposable Pasteur pipette and cut off a 0.5 inch section from the constricted tip. Insert a filter paper disk at the top of the tube, 2.5 cm from the constriction. Add a sufficient quantity of PX-21 Carbon/Celite 545 (Prepared as described in the Reagent section of this protocol) to the tube to form a 2 cm length of the Carbon-Celite. Insert a glass wool plug. Pre-elute the column in sequence with 2 mL of 50% benzene-in-ethyl acetate, 1 mL of 50% methylene chloride-in-cyclohexane and 2 mL of hexane, and discard these eluates. Load the extract (reconstituted in 1 mL of hexane) from Step h. onto the top of the column, along with 1 mL hexane rinse. Elute the column with 2 mL of 50% methylene chloride-in-hexane and 2 mL of 50% benzene-in-ethyl acetate and discard these eluates. Invert the column and reverse elute it with 4 mL of toluene, retaining this eluate for CDD/CDF analysis.

j. Concentrate each of the retained fractions to a volume of approximately 1 mL by heating the tubes in a water bath while passing a stream of prepurified N_2 over the solutions, as described above. Quantitatively transfer the concentrated fractions into separate micro-reaction vessels for the appropriate analysis. Evaporate the solutions in each of the micro-reaction vessels almost to dryness, using the procedures just mentioned, rinse the walls of each vessel down with 0.5 mL CH_2Cl_2, and reconcentrate just to dryness.

k. Approximately 1 hour before gas chromatographic-mass spectrometric (GC-MS) analysis, dilute the residue in each micro-reaction vessel with an appropriate quantity of tridecane (depending upon the anticipated quantities of analytes in each vessel) and gently swirl the solvent in the vessel to ensure dissolution of CDDs/CDFs. Inject an appropriate aliquot of this solution into the GC-MS instrument.

7.2 ANALYSIS OF SAMPLE EXTRACTS FOR PCDD/PCDF USING COUPLED GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC-MS)

Sample extracts prepared by the procedures described in the foregoing are analyzed by GC-MS utilizing the following instrumental parameters. Typically, 1 to 5 µl portions of the extract are injected into the GC. Sample extracts are analyzed for the concentrations of total tetra- through octa-CDDs and CDFs, and for 2,3,7,8-TCDD, and 2,3,7,8-TCDF.

7.2.1. Gas Chromatograph

a. Injector: Configured for capillary column, splitless/split injection (split flow on 60 seconds following injection), injector temperature, 250°C.

b. Carrier gas: Hydrogen, 30 lb head pressure

c. Capillary Column: For total tetra- through octa- CDDs/CDFs and 2,3,7,8-TCDD, 60 M x 0.25 mm I.D. fused silica DB-5; temperature, programmed, see Table 1 for temperature program.

d. Interface Temperature: 250°C

7.2.2. Mass Spectrometer

a. Ionization Mode: Electron impact (70 eV)

b. Static Resolution: 1:600 (10% valley) or 1:10,000 depending upon requirements.

c. Source Temperature: 250°C

d. Ions Monitored: Computer-Controlled Selected-Ion-Monitoring, See Table 1 for list of ion masses monitored and time intervals during which ions characteristic of each class of PCDD and PCDF are monitored.

7.2.3 Calibration Procedures

a. Calibrating the MS Mass Scale: Perfluoro Kerosene is introduced into the MS, in order to calibrate the mass scale through at least m/z 500. The mass calibration is rechecked at least at 8 hr. operating intervals.

b. Table 1A shows the GC temperature program typically used to resolve each chlorinated class of PCDD and PCDF from the other chlorinated classes, and indicates the corresponding time intervals during which ions indicative of each chlorinated class are monitored by the MS. This temperature program and ion monitoring time cycle were established by injecting aliquots of Standard Mixtures A and B. (See below for list of calibration standard mixtures). Corresponding data was established for the PCBs by injecting Standard Mixture D.

c. Checking GC Column Resolution for 2,3,7,8-TCDD. Utilize the column-resolution TCDD isomer mixtures (Standard Mixture C) to verify that 2,3,7,8-TCDD is separated from the other TCDD isomers. A 20% valley or less must be obtained between the mass chromatographic peak observed for 2,3,7,8-TCDD and adjacent peaks arising from other TCDD isomers.

d. Calibration of the GC-MS-DS system to accomplish quantitative analysis of 2,3,7,8-TCDD and 2,3,7,8-TCDF, and of the total tetra- through octa-CDDs and CDFs contained in the sample extract is accomplished by analyzing a series of at least three working calibration standards. Each of these standards is prepared to contain the same concentration of each of the stable-isotopically labelled internal standards used here (Standard Mixture A) but a different concentration of native PCDD/PCDF (Standard Mixture B). Typically, mixtures will be prepared so that the ratio of native PCDD and PCDF to isotopically-labelled PCDD and PCDF will be on the order of 0.1, 0.5 and 1.0 in the three working calibration mixtures. The actual concentrations of both native and isotopically-labelled PCDD and PCDF in the working calibration standards will be selected on the basis of the concentrations to be measured in the actual sample extracts. Equations for calculating relative response factors from the calibration data derived from the calibration standard analyses, and for calculating the recovery of the $^{13}C_{12}$-2,3,7,8-TCDD and the other isotopically-labelled PCDD and PCDF, and the concentration of native PCDD and PCDF in the sample (from the extract analysis) are summarized below. In these calculations, as can be seen, 2,3,7,8-TCDD is employed as the illustrative model. However, the calculations for each of the other native dioxins and furans in the sample analyzed are accomplished in an analogous manner. It should be noted that in view of the fact that stable-isotopically labelled internal standards corresponding to each tetra- through octachlorinated class are not used here (owing to limited availability at this time) the following approach is adopted: For quantitation of tetrachlorinated dibenzofurans $^{13}C_{12}$-2,3,7,8-TCDF is used as the internal standard. For quantitation of tetrachlorodibenzo-p-dioxins, $^{13}C_{12}$-2,3,7,8-TCDD is used as the internal standard. For quantitation of PeCDD, HxCDD, PeCDF, and HxCDF, the labelled TCDD and TCDF standards, respectively, are used. For quantitation of HpCDD, OCDD, and HpCDF, OCDF, the isotopically-labelled OCDD is used. Inherent in this approach is the assumption that the response factors for each of the isomers of each chlorinated class are equal.

7.2.4. Calibration Standard Mixtures

a. Standard Mixture A:
0.4ng/µl $^{37}Cl_4$-2,3,7,8-TCDD
0.4ng/µl $^{37}Cl_4$-2,3,7,8-TCDF
1.0ng/µl $^{13}C_{12}$-2,3,7,8-TCDD
1.0ng/µl $^{13}C_{12}$-OCDD

b. Standard Mixture B: i) 10 ng/µl of each of:
2,3,7,8-TCDD
1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDD
1,2,3,4,6,7,8-HpCDD

OCDD
2,3,7,8-TCDF
2,3,4,7,8-PeCDF
1,2,3,6,7,8-HxCDF
1,2,3,4,6,7,8-HpCDF
OCDF

ii) 2ng/µl of each of same isomers as in 4.b.i

iii) 0.4ng/µl of each of same isomers as in 4.a

c. Standard Mixture C: EPA TCDD Column Performance Mixture

7.2.5 Equations for Calculating Response Factors, Concentration of 2,3,7,8-TCDD In An Unknown Sample, and Recoveries of Internal Standards

Equation 1: Response Factor (RRF) for native 2,3,7,8-TCDD using $^{13}C_{12}$-2,3,7,8-TCDD as an internal standard.

$$RRF_d = (A_sC_{is}/A_{is}C_s)$$

where: A_s = SIM response for 2,3,7,8-TCDD ion at m/z 320 + 322

A_{is} = SIM response for $^{13}C_{12}$-2,3,7,8-TCDD internal standard ion at m/z 332

C_{is} = Concentration of the internal standard (pg./µL.)

C_s = Concentration of the 2,3,7,8-TCDD (pg./µL.)

Equation 2: Response Factor (RRF) for $^{37}Cl_4$-2,3,7,8-TCDD, the co-injected external standard

$$RRF_f = (A_{is}C_{es})/(A_{es}C_{is})$$

where: A_{is} = SIM response for $^{13}C_{12}$-2,3,7,8-TCDD internal standard ion at m/z 332

A_{es} = SIM response for co-injected $^{37}Cl_4$-2,3,7,8-TCDD external standard at m/z 328 - 0.009 (SIM response for native 2,3,7,8-TCDD at m/z 322)

C_{is} = Concentration of the internal standard (pg./µL.)

C_{es} = Concentration of the external standard (pg./µL.)

Equation 3: Calculation of concentration of native 2,3,7,8-TCDD using $^{13}C_{12}$-2,3,7,8-TCDD as internal standard

Concentration, pg./g. = $(A_s)\ (I_s)/(A_{is})(RRF_d)(W)$

where: A_s = SIM response for 2,3,7,8-TCDD ion at m/z 320 + 322

A_{is} = SIM response for the $^{13}C_{12}$-2,3,7,8-TCDD internal standard ion at m/z 332

I_s = Amount of internal standard added to each sample (pg.)

W = Weight of soil or waste in grams

RRF_d = Relative response factor from Equation 1

Equation 4: Calculation of % recovery of $^{13}C_{12}$-2,3,7,8-TCDD internal standard

% Recovery = $100(A_{is})(E_s)/(A_{es})(I_i)(RRF_f)$

A_{is} = SIM response for $^{13}C_{12}$-2,3,7,8-TCDD internal standard ion at m/z 332

A_{es} = SIM response for $^{37}Cl_4$-2,3,7,8-TCDD external standard ion at m/z 328 - 0.009 (SIM Response for native 2,3,7,8-TCDD at m/z 322)

E_s = Amount of $^{37}Cl_4$-2,3,7,8-TCDD external standard co-injected with sample extract (ng.)

I_i = Theoretical amount of $^{13}C_{12}$-2,3,7,8-TCDD internal standard in injection

RRF_f = Relative response factor from Equation 2

As noted above, procedures similar to these are applied to calculate analytical results for all of the other PCDD/PCDF determined in this method.

7.2.6 Criteria Which GC-MS Data Must Satisfy for Identification of PCB and PCDD/PCDF in Samples Analyzed and Additional Details of Calculation Procedures.

In order to identify specific PCDD/PCDF and PCB in samples analyzed, the GC-MS data obtained must satisfy the following criteria:

a. Mass spectral responses must be observed at both the molecular and fragment ion masses corresponding to the ions indicative of each chlorinated class of PCDD/PCDF and PCB identified (see Tables 1A & 1B) and intensities of these ions must maximize essentially simultaneously (within ± 1 second). In addition, the chromatographic retention times observed for each PCDD/PCDF signal must be correct relative to the appropriate stable-isotopically labelled internal standard and must be consistent with the retention time windows established for the chlorinated group to which the particular PCDD/PCDF is assigned.

b. The ratio of the intensity of the molecular ion $(M)^+$ signal to that of the $(M+2)^+$ signal must be within ± 10% of the theoretically expected ratio (for example, 0.77 in the case of TCDD; therefore the acceptable range for this ratio is 0.62 to 0.92).

c. The intensities of the ion signals are considered to be detected if each exceeds the baseline noise by a factor of at least 3:1. The ion intensities are considred to be quantitatively measurable if each ion intensity exceeds the baseline noise by a factor of at least 5:1[a].

d. For reliable detection and quantitation of PCDF it is also desirable to monitor signals arising from chlorinated diphenyl ethers which, if present could give rise to fragment ions yielding ion masses identical to those monitored as indicators of the PCDF. Accordingly, in Table 1A, appropriate chlorinated diphenyl ether masses are specified which must be monitored simultaneously with the PCDF ion-masses. Only when the response for the diphenyl ether ion mass is not detected at the same time as the PCDF ion mass can the signal obtained for an apparent PCDF be considered unique.

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[a] In practice, the analyst can estimate the baseline noise by measuring the extension of the baseline immediately prior to each of the two mass chromatographic peaks attributed to a given PCDD/PCDF. Spurious signals may arise either from electronic noise or from other organic compounds in the extract. Since it may be desirable to evaluate the judgement of the analyst in this respect, copies of original mass chromatograms must be included in the report of analytical results.

e. Measurement of the concentration of the congeners in a chlorinated class using the methods described herein is based on the assumption that all of the congeners are identical to the calibration standards employed in terms of their respective chemical and separation properties and in terms of their respective gas chromatographic and mass spectrometric responses. Using these assumptions, for example, the $^{13}C_{12}$-2,3,7,8-TCDD internal standard is utilized as the internal calibration standard for all of the 22 TCDD isomers or congeners. Furthermore, the concentration of the total TCDD present in a sample extract is determined by calculating, on the basis of the standard procedure outlined above, the concentration of each TCDD isomer peak (or peaks for multiple TCDD isomers, where these coelute) and these individual concentrations are subsequently summed to obtain the concentration of "total" TCDD. Similar procedures are applied, of course for all the other PCDD/PCDF.

f. Frequently, during the analysis of actual sample extracts, extraneous compounds which are present in the extract (those organic compounds not completely removed during the clean-up phase of the analysis) can cause changes in the liquid and gas chromatographic elution characteristics of the PCDD/PCDF (typically retention times for the PCDD/PCDF are prolonged). Such extraneous organic compounds, when introduced into the mass spectrometer source may also result in a decrease in the sensitivity of the MS because of suppression of ionization, and other affects such as charge transfer phenomena. The shifts in chromatographic retention times are usually general shifts, that is, the relative retention times for the PCDD/PCDF are not changed, although the entire elution time scale is prolonged. The analyst's intervention in the GC-MS operating sequence can correct for the lengthened GC retention times which are sometimes observed due to the presence of extraneous organics in the sample extract. For example, using the program outlined in Table 1, if the retention time observed for 2,3,7,8-TCDD (which normally is 19.5 minutes) is lengthened by 30 seconds or more, appropriate adjustments in the programming sequence outlined in Table 1 can be made, that is, each selected ion-monitoring program is delayed by a length of time proportionate to the lengthening of the retention time for the 2,3,7,8-TCDD isomer. In the case of ionization suppression, this phenomenon is inherently counteracted by the internal standard approach. However, if loss of sensitivity due to ionization suppression is severe, additional clean-up of the sample extract may be required in order to achieve the desired detection limits.

7.2.7 Quality Assurance/Quality Control

Quality assurance and quality control are ensured by the following provisions:

a. Each sample analyzed is spiked with stable isotopically labelled internal standards, prior to extraction and analysis. Recoveries obtained for each of these standards should typically be in the range from 60-90%. Since these compounds are used as true internal standards however, lower recoveries do not necessarily invalidate the analytical results for native PCDD/PCDF or PCB but may result in higher detection limits that are desired.

b. Processing and analysis of at least one method blank sample is accomplished for each set of samples (a set being defined as 20 samples or less). Analyses of field and travel blanks may also be desirable.

7.3 REAGENTS AND CHEMICALS

The following reagents and chemicals are appropriate for use in the procedures described above. In all cases, equivalent materials from other suppliers may also be used.

7.3.1 Sources of Chemicals, Procedures Employed for Preparing Reagents

a. Potassium Hydroxide, Anhydrous, Granular Sodium Sulfate and Sulfuric Acid (all Reagent Grade): J.T. Baker Chemical Co. or Fisher Scientific Co. The granular sodium sulfate is purified prior to use by placing a beaker containing the sodium sulfate in a 400°C oven for four hours, then removing the beaker and allowing it to cool in a desiccator. Store the purified sodium sulfate in a bottle equipped with a Teflon-lined screw cap.

b. Hexane, Methylene Chloride, Benzene, Methanol, Toluene, Isooctane: "Distilled in Glass" Burdick and Jackson.

c. Tridecane (Reagent Grade): Sigma Chemical Co.

d. Basic Alumina (Activity Grade 1, 100 - 200 mesh): ICN Pharmaceuticals. Immediately prior to use, the alumina is activated by heating for at least 16 hours at 600°C in a muffle furnace and then allowed to cool in a dessicator for at least 30 minutes prior to use. Store preconditioned alumina in a desiccator.

e. Silica (Bio-Sil A, 100/200 mesh): Bio-Rad. The following procedure is recommended for conditioning the Bio-Sil A prior to use. Place an appropriate quantity of Bio-Sil A in a 30 mm x 300 mm long glass tube (the silica gel is held in place by glass wool plugs) which is placed in a tube furnace. The glass tube is connected to a pre-purified nitrogen cylinder, through a series of four traps (stainless steel tubes, 1.0 cm O.D. x 10 cm long)[6]: 1) Trap No. 1 - Mixture comprised of Chromosorb W/AW (60/80 mesh coated with 5% Apiezon L), Graphite (UCP-1-100), Activated Carbon (50 to 200 mesh) in a 7:1.5:1.5 ratio (Chromosorb W/AW, Apiezon L obtained from Supelco, Inc., Graphite obtained from Ultracarbon Corporation, 100 mesh, 1-M-USP; Activated Carbon obtained from Fisher Scientific Co.; 2) Trap No. 2 - Molecular Sieve 13 X (60/80 mesh), Supelco, Inc.; 3) Trap No. 3 - Carbosieve S (80/100 mesh), obtained from Supelco, Inc.; 4) The Bio-Sil A is heated in the tube for 30 minutes at 180°C while purging with nitrogen (flow rate 50-100 mL/minute), and the tube is then removed from the furnace and allowed to cool to room temperature. Methanol (175 mL) is then passed through the tube, followed by 175 mL methylene chloride. The tube containing the silica is then returned to the furnace, the nitrogen purge is again established (50-100 mL flow) and the tube is heated at 50°C for 10 minutes, then the temperature is gradually increased to 180°C over 25 minutes and then maintained at 180°C for 90 minutes. Heating is then discontinued but the nitrogen purge is continued until the tube cools to room temperature. Finally, the silica is transferred to a clean, dry, glass bottle and capped with a Teflon-lined screw cap for storage.

f. Silica Gel Impregnated With Sulfuric Acid: Concentrated sulfuric acid (44 g) is combined with 100 g Bio-Sil A (conditioned as described above) in a screw capped bottle and agitated to mix thoroughly. Aggregates are dispersed with a stirring rod until a uniform mixture is obtained. The H_2SO_4-silica gel is stored in a screw-capped bottle (Teflon-lined cap).

g. Silica Gel Impregnated with Sodium Hydroxide: 1N Sodium hydroxide (39 g) is combined with 100 g Bio-Sil A (conditioned as described above) in a screw capped bottle and agitated to mix throughly. Aggregates are dispersed with a stirring rod until a uniform mixture is obtained. The NaOH-silica gel is stored in a screw-capped bottle (Teflon-lined cap).

h. Carbon/Celite: Combine Amoco PX-21 carbon (10.7 g) with Celite 545 (Fisher Scientific Co.) (124 g) in a 250 mL glass bottle fitted with a Teflon-lined cap. Agitate the mixture to combine thoroughly. Store in the screw-capped bottle.

i. Nitrogen and Hydrogen (Ultra High Purity): Matheson Scientific

j. Fused Silica Capillary Gas Chromatographic Column: 60 M fused silica (0.25 mm I.D.) capillary column coated with DB-5 (0.25 µ film thickness), J & S Scientific, Inc., Crystal Lake, IL.

k. Chlorinated Dibenzo-p-dioxins and Dibenzofurans Used As Calibration Standards: $^{37}Cl_4$-2,3,7,8-TCDD (SSY-6-123) and $^{37}Cl_4$-2,3,7,8-TCDF (DF-14) were obtained from KOR, Inc. $^{13}C_{12}$-2,3,7,8-TCDD (AWN 1203-65) and $^{13}C_{12}$-OCDD (SSY-8-78) were obtained from Cambridge Isotope Laboratores. The 22 TCDD standards and all other CDDs/CDFs employed in the study were synthesized in the Brehm Laboratory. A column performance check standard was obtained from USEPA (Check Standard Mixture #2) which contained 1,4,7,8-TCDD; 2,3,7,8-TCDD; 1,2,3,4-TCDD; 1,2,3,7/1,2,3,8-TCDD; 1,2,7,8-TCDD and 1,2,6,7-TCDD.

TABLE 1

Sequence of Operations in GC-MS-DS Quantitation of CDDs/CDFs in Extracts of Environmental Samples

Elapsed Time (min)	Event	GC Column Temperature* (°C)	Temperature Program Rate (°C/min)	Ions Monitored by Mass Spectrometer (m/z)	Identity of Fragment Ion	Compounds Monitored	Approximate Theoretical Ratio of $[M]^+:[M+2]^+$
0.00	Injection, splitless	190					
1.00	Turn on split valve	190					
1.00	Begin temp. program to 220°C	190	5				
6.00	Open column flow to mass spectrometer	215	5				
7.00	Column temperature hold	220					
14.00	Start Tetra Program; sweep = 350 ppm; time/mass = 0.08 sec.			240.938	$[M-COCl]^+$	TCDF	
				258.930	$[M-COCl]^+$	TCDD	
				303.902	$[M]^+$	TCDF	0.77
				305.899	$[M+2]^+$	TCDF	
				315.942	$[M]^+$	$^{13}C_{12}$-TCDF	
				319.897	$[M]^+$	TCDD	0.77
				321.894	$[M+2]^+$	TCDD	
				327.805	$[M]^+$	$^{37}Cl_4$-TCDD	
				331.937	$[M]^+$	$^{13}C_{12}$-TCDD	
				373.840	$[M]^+$	HxDPE$^{a.}$	
22.00	Stop Tetra Program	220					
22.50	Start Penta Program; sweep = 350 ppm; time/mass = 0.12 sec.			274.899	$[M-COCl]^+$	PeCDF	
				290.894	$[M-COCl]^+$	PeCDD	
				337.863	$[M]^+$	PeCDF	1.54
23.00	Begin temp. program to 235°	220	5	339.860	$[M+2]^+$	PeCDF	
26.00	Column temperature hold	235		353.858	$[M]^+$	PeCDD	1.54
				355.855	$[M+2]^+$	PeCDD	
				407.801	$[M]^+$	HpDPE$^{a.}$	
32.00	Stop Penta Program						
32.50	Start Hexa Program; sweep = 350 ppm; time/mass = 0.20 sec.	235		310.857	$[M-COCl]^+$	HxCDF	
				326.852	$[M-COCl]^+$	HxCDD	
				373.821	$[M]^+$	HxCDF	1.23
				375.821	$[M+2]^+$	HxCDF	
				385.861	$[M]^+$	$^{13}C_{12}$-HxCDF	
				389.816	$[M]^+$	HxCDD	1.23
				391.813	$[M+2]^+$	HxCDD	
				411.856	$[M]^+$	$^{13}C_{12}$-HxCDD	
				443.759	$[M]^+$	ODPE$^{a.}$	

TABLE 1 (continued)

Sequence of Operations in GC-MS-DS Quantitation of CDDs/CDFs in Extracts of Environmental Samples (Cont.)

Elapsed Time (min)	Event	GC Column Temperature (°C)	Temperature Program Rate (°C/min)	Ions Monitored by Mass Spectrometer (m/z)	Identity of Fragment Ion	Compounds Monitored	Approximate Theoretical Ratio of $[M]^+:[M+2]^+$
33.00	Begin temp. program to 250°C	235	5				
36.00	Column temperature hold	250					
42.50	Stop Hexa Program						
43.00	Start Hepta Program; sweep = 350 ppm; time/mass = 0.30 sec.	250		344.818	$[M-COCl]^+$	HpCDF	
				360.813	$[M-COCl]^+$	HpCDD	
				407.782	$[M]^+$	HpCDF	1.03
				409.779	$[M+2]^+$	HpCDF	
				423.777	$[M]^+$	HpCDD	1.03
				425.774	$[M+2]^+$	HpCDD	
				477.720	$[M^+]$	NDPE[a].	
53.00	Stop Hepta Program	250					
53.50	Start Octa Program; sweep = 350 ppm; time/mass = 0.30 sec.	250		378.768	$[M-COCl]^+$	OCDF	
				394.774	$[M-COCl]^+$	OCDD	
54.00	Begin temp. program to 270°	250*	5	441.732	$[M]^+$	OCDF	0.88
58.00	Column temperature hold	270*		443.740	$[M+2]^+$	OCDF	
				453.772	$[M]^+$	$^{13}C_{12}$-OCDF	
				457.738	$[M]^+$	OCDD	0.88
				459.735	$[M+2]^+$	OCDD	
				469.779	$[M]^+$	$^{13}C_{12}$-OCDD	
				471.776	$[M+2]^+$	$^{13}C_{12}$-OCDD	
65.00	Stop Octa Program			511.681	$[M]^+$	DDPE[a].	
65.00	Begin temp. program to 300°	270*	5				
71.00	Column temperature hold	300*					
75.00	Cool Column to 190°						

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[a] HxDPE, HpDPE, ODPE, NDPE, DDPE are abbreviations which designate (respectively) hexachloro-, heptachloro-, octachloro-, nonachloro-, and decachlorodiphenyl ethers.

*The parameters given here are applicable for a 60-meter fused silica capillary GC column coated with DB-5.

APPENDIX D

INCINERATOR EXHAUST STUDY SAMPLING RESULTS

APPENDIX D

I. ORGANIZATION OF DATA

The analytical results of the Dow Chemical Company Midland Plant Building 703 incinerator emissions study encompass a wide variety of influent and effluent streams, analyzed for the following generalized categories of compounds:

- Volatile compounds, or those with boiling points generally below 100°C,
- Semi-volatile compounds, with boiling points greater than 100°C, and
- PCDD/PCDF. These were analyzed separately from other semi-volatile compounds, as described below.

In addition, incinerator exhaust gases were sampled for vinylidene chloride using a direct capture method with immediate instrumental analysis, as the analytical methods for other volatile compounds were not amenable to vinylidene chloride. Further detail concerning these analyses are contained in Appendix A, Section III.C. of this report.

In general, the data are presented below individually for each type of stream, and in terms of volatile compounds, semi-volatile compounds, and PCDD/PCDF, in that order. Discussion of quality assurance aspects relating to each category of stream and compound group is presented to highlight the information contained in the data tables.

II. ANALYTICAL LABORATORIES

As indicated above, PCDD/PCDF analyses were performed by an analytical laboratory other than that involved with volatile and semi-volatile compounds, owing to the comparatively limited number of capable laboratories. The Brehm Laboratory of Wright State University, Fairborn, Ohio, completed these analyses, while the EAL Corporation of Richmond, California, was selected to analyze the samples for volatile and semi-volatile compounds.

III. ANALYTICAL RESULTS

A. Acceptability

In the sections to follow, data are generally presented in tables which are based on concentration, with accompanying tables showing raw data as presented by the analytical laboratories. Either of these tables may include quality assurance data relating to accuracy (% recovery of known surrogate compounds introduced to the analyzed matrix by the laboratory).

1. PCDD/PCDF

For PCDD/PCDF, the ranges of acceptability defined in the Quality Assurance Project Plan[7] for the study were 70 to 130% recoveery for two isotopically labeled analogs ($^{13}C_{12}$ 2378-TCDD and $^{37}Cl_4$ 2378-TCDF) and 50 to 150% for two others ($^{37}Cl_4$ 2378-TCDD and $^{13}C_{12}$ OCDD). However, in comparing these acceptance criteria to those commonly used in other current work involving analyses for PCDD/PCDF, they were found to be overly stringent. In judging the acceptability of PCDD/PCDF data, therefore, a range of recoveries of 50 to 150% was considered acceptable.

The internal standard $^{13}C_{12}$ 2378-TCDD is a primary importance as the accuracy determinant for tetra- through hexa-CDD; those homologue groups are of greatest priority in assessing potential risks to health. Recoveries of the second 2378-TCDD surrogate, $^{37}Cl_4$ 2378-TCDD, serves to confirm the recoveries of $^{13}C_{12}$ 2378-TCDD. In summary, if both 2378-TCDD surrogates are recovered within the acceptable range of 50 to 150%,, the analytical data are defined as acceptable for the homologues of greatest concern.

Recoveries of the internal standard $^{13}C_{12}$ OCDD were frequently poorer than for the other standards. However, this internal standard measures analytical accuracy for hepta- and octa-CDD and CDF homologues, which are of comparatively low concern in terms of risk assessment. Recoveries of $^{37}Cl_4$ 2378-TCDF are used to judge the accuracy of tetra- through hexa-CDF data, which, with respect to risk, are of lower priority than the corresponding PCDDs.

In the PCDD/PCDF data in this Appendix, completeness is calculated and presented individually by standard. According to the above discussion, the value of the PCDD/PCDF data should be judged primarily by the accuracy of recovery of the two labeled 2378-TCDD compounds. Completeness in this area was generally near or above 80%; this performance confirms the overall validity of the analytical data in calculating general mass balances and risk assessment.

2. Other Compounds

The Quality Assurance Project Plan references ranges of acceptable surrogate recovery of 20 to 180% for semi-volatile compounds, and 80 to 125% for volatile compounds. For semi-volatile compounds, six surrogates were used -- three acid and three base-neutral, while for volatile compounds three or four surrogates were used, depending upon the type of sample. There is no currently accepted guidance relating specific surrogates to particular analytes. However, the evaluate the acceptability of semi-volatile compound analyses, if the recovery of all three acid surrogates was acceptable, then the analysis of any detected acid compound was considered valid; the same was done for base-neutral compounds. On the semi-volatile compound data tables to follow in this section and in Appendix D of this report, data which were treated in this way are appropriately labeled. To assess overall completeness, however, data were defined as valid only if all of the semi-volatile surrogate compounds were analyzed within range.

For volatile compounds, as there was no available summary of the ranges of compounds to which particular surrogates are associated, data points were considered acceptable only if the recoveries of all three or four surrogates were within the target range of 80 to 125%. Detailed inspection of the volatile compound data tables which include surrogate recovery information reveal many cases in which the recoveries of most surrogates were very close to the target range. Therefore, the volatile compound data are probably more reliable than a strict interpretation of the accuracy data would indicate.

B. Precombustion Air

1. Volatile Compounds

These data appear in Table D-1 in terms of concentration. The raw analytical data used to derive them are presented in Table D-2.

The method blank, which was comprised of 1.5 grams of Tenax GC sorbent sent directly from GCA to the analytical laboratory, EAL Corporation, showed the presence of measurable amounts of chloroform, perchloroethylene, methylcyclohexane, and 1,3-dichlorobenzene. The last two compounds were not detected in any exposed sample. However, chloroform and perchloroethylene were found at higher concentrations than in any exposed sample, indicating that both compounds were present as laboratory contamination.

Two of the eight sample sets were acceptable in terms of accuracy (% surrogate recovery, see Table D-2). Of the target volatile compounds shown in Table D-1, three,

- carbon tetrachloride (days 1 and 3)
- monochlorobenzene (days 1 and 3), and
- trichloroethylene (day 1 only),

were detected in the concentrations indicated. However, target precision criteria of $\leq 50\%$ RPD (between day 1 sample and field duplicate results) were met only for monochlorobenzene. Accuracy (surrogate recovery) data were unacceptable for samples taken on the second sampling day. Other compounds of possible interest detected only on the first sampling day included ethylbenzene and xylene (total xylenes); however, precision criteria were not met for either compound. Benzene and toluene were noted on the third sampling day, but as there was no duplicate sample taken on this day, this result is considered tentative.

Detection limit objectives of 1 ppb in air were achieved for all of the above-listed detected compounds as shown in Table D-1. Actual detection limits were in the range of 0.3 to 0.8 ppb for the compounds detected above.

These samples were obtained with a 14-day target limit for holding time prior to analysis. Samples were actually held for periods of 19 to 27 days before analysis. Thus, the results presented are considered to be conservative it is possible that some compounds may have been lost or altered due to decay or reaction in the time between sampling and analysis.

TABLE D-1
VOLATILE COMPOUNDS - PRE-COMBUSTION AIR
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84
COMPOUND CONCENTRATION (ug/m^3)^1

SAMPLING DATE	COMPOUNDS DETECTED					ACCURACY (% SURROGATE RECOVERY) (SAMPLE TUBE/FIELD BLANK TUBE)					APPROXIMATE DETECTION LIMIT IN AIR (ug/m^3)
	Carbon tetrachloride	Monochlorobenzene	Trichloroethylene	1,4-dichlorobenzene^2	Ethylbenzene	Toluene - D8	Bromofluorobenzene	1,2-dichloroethane - D4	Ethylbenzene - D10	ACCEPTABLE^3	
8/28/84	15.35	12.87	0.64	72.43	10.63	94/94	154/108	58/86	186*/108	N/Y	0.40 - 0.80
8/28/84 (Field Duplicate)	61.00	20.55	3.12	60.20	29.26	98/94	110/108	54/86	106/108	N/Y	0.37 - 0.74
8/30/84	222.50	--	16.98	81.69	--	0*/106	0*/86	0*/60	0*/110	N/N	0.37 - 0.74
9/5/84	26.69	29.04	--	45.35	--	86/100	92/116	87/78	112/104	Y/N	0.32 - 0.64
Precision (RPD) 8/28/84 Samples	120	46	132	18	93						

Notes:
1 Sample concentration less field blank concentration.
2 Compound tentatively identified.
3 All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.
* Recovery outside of acceptable range of 80-125%

TABLE D-2
QUANTITATED AND TENTATIVELY-IDENTIFIED VOLATILE COMPOUNDS DETECTED IN PRECOMBUSTION
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

		QUANTITATED COMPOUNDS												TENTATIVELY-IDENTIFIED COMPOUNDS										ACCURACY (% SURROGATE RECOVERY)				
	UNITS	Chloroform	Carbon tetrachloride	Trichloroethylene	Perchloroethylene	Monochlorobenzene	Ethylbenzene	Total xylenes	1,1,1-trichloroethane	Toluene	Acetone	2-butanone	Benzene	1,4-dichlorobenzene	Trichlorotrifluoromethane	Methylcyclopentane	Hexamethylcyclotrisiloxane	Methylcyclohexane	1,3-dichlorobenzene	3-methylpentane	1,2-dichlorobenzene	2-methyl-1-pentane	Benzaldehyde	Toluene - D8	Bromofluorobenzene	1,2-dichloroethane - D4	Ethylbenzene - D10	ACCEPTABLE[1]
8/28/84 Sample	ng	88	301	22	127	161	186	786					90	1137	6481	5590	200							94	154	58	186	No
Field Duplicate		194	872	53	182	257	419	1302	875					984							657		470	98	110	54	106	No
Field Blank			109	14	44		53	80		1291			57	231										94	108	86	108	Yes
8/30/84 Sample		54	3373	290				8455		16865	10			1192										0	0	0	0	No
Field Blank			347	59	36		22	112		559			1182	81										106	86	60	110	No
9/5/84 Sample			843		138	456			1706	962			756	1420						1390	97			86	92	87	112	Yes
Field Blank			424	41	52		295	343		626				708			200			7000		700		100	116	78	104	No
Tenax GC® Method Blank		645			403													124	4084					117	77	105	62	No

Note: [1] All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.

COMPLETENESS - 25% (2/8)

2. Semi-Volatile Compounds

The results of these analyses are reported in Tables D-3 and D-4.

The method blank, composed of 75 grams of XAD-2 sorbent, was analyzed and found free of contamination (see Table D-4). However, this sample was extracted and diluted prior to analysis, such that surrogate compounds added to the matrix were poorly detected. Since the field blank samples showed the presence only of ubiquitous phthalate compounds commonly considered laboratory-related, and these analyses were satisfactory with respect to surrogate recoveries (accuracy), it was determined that the sorbents employed in sampling were free of background quantities of several compounds of interest detected in sampled air.

As the data presented in Tables D-3 and D-4 indicate, 1,2-dichlorobenzene and 1,2,4-trichlorobenzene were found on all three sampling days; field duplicate sampling on the second day indicated precision was within the objectives of the study for these two compounds. Another dichlorobenzene, the 1,4 isomer, was also detected on all three days, but precision could not be judged as it was not found in the field duplicate. Low concentrations of 1,3-dichlorobenzene were detected on the first and third sampling days, but none on the second day, when a field duplicate was obtained.

Other target compounds were detected, as follows:

- 1-1 biphenyl (day 1),
- biphenyl (day 2, but not in field duplicate), and
- monochlorobenzene (days 1 and 2).

The latter is a volatile compound for which the previously described volatile air sampler was considered more appropriate. The precision of the analytical method for volatile compounds appeared better than that for semi-volatiles in the case of monochlorobenzene. In any event, the concentrations of monochlorobenzene measured by both methods were comparable within an order of magnitude.

Naphthalene was detected on all three sampling days, but satisfactory precision was not achieved, as measured in the field duplicate sample on the second day. Several substituted benzenes were seen on all three days, with a host of isomers in comparatively high concentrations observed on the first day.

The target detection limit criterion of 5 ppb in air for semi-volatile compounds was achieved; actual detection limits, for 1,2,4-trichlorobenzene, for example, were on the order of 0.05 ppb. Accuracy criteria (20 to 180% surrogate recovery) were met for seven of the eight samples, including field and method blanks and duplicates (see Tables D-3 and D-5).

A summary assessment of these data indicates that while a wide variety of semi-volatile compounds were detected, the presence of only two, 1,2-dichlorobenzene, and 1,2,4-trichlorobenzene, could be established and supported by acceptable measures of accuracy. The presence of other compounds should be considered a tentative finding.

TABLE D-3
SEMI-VOLATILE COMPOUNDS - PRE-COMBUSTION AIR
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84
COMPOUND CONCENTRATION (ug/m^3)^1

	TARGETED COMPOUNDS								OTHER COMPOUNDS DETECTED								ACCURACY (% SURROGATE RECOVERY) (SAMPLE TUBE/FIELD BLANK TUBE)						
																	Base-Neutrals			Acids			
SAMPLING DATE	Monochlorobenzene	1,2-dichlorobenzene	1,3-dichlorobenzene	1,4-dichlorobenzene	1,2,4-trichlorobenzene	Naphthalene	1,1'-biphenyl^2	Biphenyl	2-methylnaphthalene	Ethylbenzene^2	1-ethyl-2-methylbenzene^2	1-(methylethyl)-benzene^2	1,2-diethylbenzene^2	1,3-diethylbenzene^2	1,2-diethenylbenzene^2	Diphenylether	Nitrobenzene - D5	2-fluorobiphenyl	Terphenyl - D14	Phenol - D5	2-fluorophenol	2,4,6-tribromophenol	Acceptable^3
8/28/84	3.08	1.42	0.15	1.41	1.58	0.44	2.22	--		2.19	2.41	1.92	2.78	1.26	0.96	4.74	94/63	95/76	142/148	87/84	101/80	75/41	YES
8/30/84	0.53	0.84	--	0.74	0.86	0.08	--	0.90		0.50							67/85	59/74	112/116	36/33	31/34	53/55	YES
8/30/84 Field Duplicate	1.84	1.03	--	--	1.19	0.64	--	--	0.25								96/85	65/74	122/116	49/33	45/34	59/55	YES
9/5/84	--	3.73	0.07	3.24	2.59	1.23	--	--	1.65								104/98	61/58	58/98	88/79	90/85	80/48	YES
																	Completeness = 8/8 = 100%						
Precision (RPD) 8/30/84 Samples	111	21	--	--	32	156																	

Notes:
1 Sample concentration less field blank concentration.
2 Compound tentatively identified.
3 All surrogate recoveries (sample and field blank) within target range (20-180%) established in Quality Assurance Project Plan.

TABLE D-4
QUANTITATED AND TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS DETECTED IN PRECOMBUSTION AIR
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

	1,2-dichlorobenzene	1,3-dichlorobenzene	1,4-dichlorobenzene	1,2,4-trichlorobenzene	Naphthalene	Dimethylphthalate	Anthracene	Di-n-butyl-phthalate	Di-n-octyl-phthalate	Phenanthrene	Bis (2-ethylhexyl)-phthalate	2-methylnaphthalene	Benzene-carbothoic acid	Monochlorobenzene
8/28/84														
Sample	5889	613	5828	6564	1840	613	159	1754	2742				12526	12772
Field Blank								3170	4243					
8/30/84														
Sample	3470		3058	3555	330			1907	6040	219	1127			2180
Field Duplicate	3592	252		4167	2241				1954	459	890	890		6414
Field Blank								3295	6300		3121			
9/5/84														
Sample	14271		12406	9050	4711			3593	3457	267	1355	6338		
Field Blank								2677	6813		1898			
Method Blank	(NO COMPOUNDS DETECTED - SURROGATE RECOVERIES UNACCEPTABLE)													

	Ethylbenzene	1,3,5,7-cycloocta-tetraene	1-(methylethyl)-benzene	1-ethyl-2-methyl-benzene	Decane	Octamethyl-cyclotetrasiloxane	1,2-diethylbenzene	1,3-diethylbenzene	1,2-diethenylbenzene	1,1-biphenyl	Diphenylether	Bis(2-methylpropyl) phthalate	Xylene	Propylbenzene
8/28/84														
Sample	9044	16955	7956	9998	14298	8963	11526	5207	3978	9209	19647	23914		
Field Blank														
8/30/84														
Sample	2082										3031		2283	930
Field Duplicate	5644		2309											3848
Field Blank														
9/5/84														
Sample					16222						12000	3666		
Field Blank														
Method Blank														

	2,4-dimethyl-2,3-heptadiene-5-yne	Diethylbenzene	Undecane	2-ethyl-1,4-dimethyl-benzene	Dodecane	Biphenyl	Methyldiphenyl-silane	Butylmethyl-propylphthalate	3,3,5-trimethylheptane	2-ethyl-1,1'-biphenyl	2-butoxy-dthanol	2,6-dimethyl-octane	Tridecane	Pentadecane
8/28/84														
Sample														
Field Blank														
8/30/84														
Sample	1916	929	2260	895	2389	3738	24121	2632						
Field Duplicate						7697	5644	890	3335	1282				
Field Blank														
9/5/84														
Sample	30853			13062							8921	2928	13200	6000
Field Blank														
Method Blank														

NOTE - Results stated in ug/kg.

TABLE D-5

QUALITY ASSURANCE DATA - PRECOMBUSTION AIR SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

	ACCURACY (% SURROGATE RECOVERY)							
	Base-Neutrals			Acids				
	Nitrobenzene-D5	2-fluorobiphenyl	Terphenyl-D14	Phenol - D5	2-fluorophenol	2,4,6-tribromophenol		ACCEPTABLE[1]
8/28/84								
SAMPLE	94	95	142	87	101	75		YES
FIELD BLANK	63	76	148	84	80	41		YES
8/30/84								
SAMPLE	67	59	112	36	31	53		YES
FIELD DUPLICATE	96	65	122	49	45	59		YES
FIELD BLANK	85	74	116	33	34	55		YES
9/5/84								
SAMPLE	104	61	58	88	90	80		YES
FIELD BLANK	98	58	98	79	85	48		YES
METHOD BLANK	12	0	6	100	28	0		NO

Completeness[2]	Base-Neutrals	Acids	Overall
	88%	88%	88% (7/8)

Notes: [1] Recoveries of all surrogate compounds within target range (20 to 180%) established in Quality Assurance Project Plan.

[2] By class of surrogates (acid and base-neutral(s)) and overall (combined).

3. PCDD/PCDF

a. All Homologues

In Table D-7 analytical data are presented in terms of weight per sample; these data are expressed in units of concentration in Table D-6. The data are self-explanatory; note that for the two homologues detected in both samples (actual and field duplicate) on August 28, the precision criterion (50% RPD or less) was met for both. However, accuracy criteria were met for only one of the four surrogates. Field blank samples were free of detectable PCDD/PCDF, with accuracies as shown.

In summary, while OCDD and TCDF were detected on the first sampling day, the accuracy of quantification is questionable as the recovery of surrogate compounds was unacceptable. These and other homologues were found on the other sampling days, but accuracy was unacceptable on the second sampling day, and precision was not determined on the third sampling day. Accuracy criteria, however, were met on the third sampling day.

b. TCDD Isomers

These data are shown in raw form in Table D-9, and expressed as concentrations in Table D-8. On the first sample day, TCDD was found only as the 1368 and 1379 isomers, while on the second day a wider diversity of isomers was detected, including the only finding of the 2378 isomer in any sample obtained in this study. The third sample day also showed a comparatively diverse range of isomers.

As for all of the TCDD isomer analyses conducted during this study, no accuracy data are stated, as no surrogate isomers were added to the analyzed matrices. The precision and accuracy limitations stated above for the analyses of all homologues should also be applied to these data.

C. Liquid Waste Feeds

1. Concentrated Liquid Wastes

a. Volatile Compounds

These data are shown in Table D-10. Substantial analytical problems were encountered with these samples; some of these are apparent in scanning the surrogate recovery data shown in these tables. Other problems with individual data are described in the notes included in the tables. Generally, however, internal quality assurance review of the volatile pollutant data revealed that they should be used with caution, as they showed a high level of contamination of column degradation material. As a result of delays in preparing sample extracts, volatile organic analyses were not performed until at least four months after the samples were first obtained. Surrogate recoveries for four data points (see above-referenced table) were out of acceptable ranges owing to dilutions necessary to respond to peak saturation problems. Calibration checks

TABLE D-6

INCINERATOR PRECOMBUSTION AIR - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

													Accuracy (%Surrogate Recovery)			
SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	HpCDF	Total OCDF	$^{13}C_{12}$2378-TCDD	$^{37}Cl_4$2378-TCDD	$^{13}C_{12}$OCDD	$^{37}Cl_4$2378-TCDF
8/28/84																
MODIFIED METHOD 5 TRAIN	ND (7.86)	58.21	ND (11.01)	ND (6.62)	ND (12.02)	216.60	ND (7.89)	391.22	ND (6.07)	ND (16.2)	ND (27.50)	21.18	84	85	17	100
FIELD DUPLICATE	ND (11.32)	ND (53.4)	ND (131)	ND (125)	ND (5.43)	335.14	ND (29.2)	628.02	ND (6.01)	ND (4.20)	ND (8.45)	ND (30.2)	2	125	22	100
FIELD BLANK				(Sample analysis not returned from laboratory.)												
8/30/84																
MODIFIED METHOD 5 TRAIN	5.16	17.99	ND (2.30)	10.39	235.10	802.08	12.93	12.93	12.50	14.23	108.48	113.67	99	92	35	100
FIELD BLANK													100	90	27	48
9/5/84																
MODIFIED METHOD 5 TRAIN	ND (1.48)	38.90	ND (0.94)	ND (1.46)	98.14	306.51	ND (1.74)	206.60	ND (1.45)	ND (1.42)	37.43	30.95	89	92	61	100
FIELD BLANK	ND (0.55)	ND (0.35)	ND (0.40)	ND (0.85)	ND (2.15)	ND (4.83)	ND (0.39)	ND (0.29)	ND (0.37)	ND (0.33)	ND (3.08)	ND (4.21)	77	97	59	76
									COMPLETENESS BY SURROGATE				71%	86%	29%	71%

Notes: Data expressed in pg/m^3.
1 All surrogate recoveries within target ranges of 50-150%.

TABLE D-7
INCINERATOR PRECOMBUSTION AIR - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	TOTAL TCDD	TOTAL PeCDD	TOTAL HxCDD	TOTAL HpCDD	OCDD	2378-TCDF	TOTAL TCDF	TOTAL PeCDF	TOTAL HxCDF	TOTAL HpCDF	OCDF
8/28/84 Hi-Vol Filter + XAD-2 sorbent	ND(2.47)	18.3	ND(3.46)	ND(2.08)	ND(3.78)	68.1	ND(2.48)	123	ND(1.60)	ND(4.27)	ND(7.25)	6.66
Field Blank				(Sample analysis not returned from laboratory.)								
Field Duplicate	ND(3.75)	ND(17.7)	ND(43.4)	ND(41.5)	ND(1.80)	111	ND(9.67)	208	ND(1.99)	ND(1.39)	ND(2.80)	ND(10.0)
8/30/84 Hi-Vol Filter + XAD-2 sorbent	1.59	5.54	ND(0.709)	3.20	72.4	247	3.98	3.98	3.85	4.38	33.4	35.0
Field Blank	ND(0.237)	ND(0.129)	ND(0.668)	ND(1.13)	ND(1.39)	ND(3.65)	ND(0.342)	ND(0.371)	ND(0.603)	ND(1.01)	ND(1.60)	ND(4.29)
9/5/84 Hi-Vol Filter + XAD-2 sorbent	ND(0.501)	13.2	ND(0.318)	ND(0.496)	33.3	104	ND(0.590)	70.1	ND(0.492)	ND(0.483)	12.7	10.5
Field Blank	ND(0.187)	ND(0.120)	ND(0.135)	ND(0.287)	ND(0.725)	ND(1.63)	ND(0.132)	ND(0.0973)	ND(0.124)	ND(0.110)	ND(1.04)	ND(1.42)

NOTE: Data expressed in ng/g.

TABLE D-8
INCINERATOR PRECOMBUSTION AIR - TCDD ISOMER ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
8/28/84															
MODIFIED METHOD 5 TRAIN	44.21	13.99													
FIELD DUPLICATE															
8/30/84															
MODIFIED METHOD 5 TRAIN		4.32		1.62				0.97	0.81	5.03	5.16				
9/5/84															
MODIFIED METHOD 5 TRAIN	23.96	7.57		2.45					0.98	3.92					

Note - Data expressed in pg/m^3.

TABLE D-9
INCINERATOR PRECOMBUSTION AIR - TCDD ISOMERS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
8/28/84															
Hi-Vol Filter + XAD-2 Sorbent	13.9	4.40	ND(2.31)	ND(2.70)	ND(2.70)	ND(2.70)	ND(2.70)	ND(1.16)	ND(2.70)	ND(2.70)	ND(2.44)	ND(1.54)	ND(1.54)	ND(1.54)	ND(1.54)
Field Blank	(Sample analysis not returned from laboratory.)														
Field Duplicate	ND(247)	ND(212)	ND(141)	ND(70.7)	ND(17.7)	ND(17.7)	ND(17.7)	ND(17.7)	ND(17.7)	ND(17.7)	ND(3.75)	ND(17.7)	ND(17.7)	ND(17.7)	ND(17.7)
8/30/84															
Hi-Vol Filter + XAD-2 Sorbent	ND(0.204)	1.33	ND(0.204)	0.500	ND(0.204)	ND(0.204)	ND(0.204)	0.300	0.250	1.55	1.59	ND(0.163)	ND(0.196)	ND(0.244)	ND(0.204)
Field Blank	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.237)	ND(0.129)	ND(0.129)	ND(0.129)	ND(0.129)
9/5/84															
Hi-Vol Filter + XAD-2 Sorbent	8.13	2.57	ND(0.611)	0.830	ND(0.611)	ND(0.611)	ND(0.611)	ND(0.611)	0.332	1.33	ND(0.501)	ND(0.611)	ND(0.611)	ND(0.611)	ND(0.611)
Field Blank	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.187)	ND(0.120)	ND(0.120)	ND(0.120)	ND(0.145)

NOTE: Data expresse1 in ng/g.

TABLE D-10
QUANTITATED VOLATILE COMPOUNDS - LIQUID WASTE INPUTS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

	TARGET COMPOUNDS			OTHER CHLORINATED COMPOUNDS			BENZENE RING COMPOUNDS					OTHER COMPOUNDS				ACCURACY (% SURROGATE RECOVERY)			
	Monochlorobenzene	Carbon tetrachloride	Chloroform	Methylene chloride	Chloromethane	Tetrachloroethylene	Benzene	Ethylbenzene	Styrene	Toluene	Total xylenes	Acetone	2-butanone	2-hexanone	Bromomethane	Toluene - D8	Bromofluorobenzene	1,2-dichloroethane-D4	ACCEPTABLE³
REAGENT BLANK 1				11			131					393	1057			88	86	96	Y
REAGENT BLANK 2					144		192									102	106	82	Y
8/28/84																			
Nozzle BA								1494				1478				104	116	84	Y
Nozzle BA Dilution	15300					7700		2050	350							(SEE 1 BELOW)			
Nozzle BB #1								35600	15900	1700	2700					106	100	86	Y
Nozzle BB #2										260					470	104	108	86	Y
Nozzle BB #2 Dilution				50			311					1700				(SEE 1 BELOW)			
Nozzle C										2370		990				76	86	84	N
Nozzle C RERUN										2110		950							
Field Blank																104	100	88	Y
8/30/84																			
Nozzle BA									35500	1950	1850			3400		100	110	86	Y
Nozzle BA Field Blank																104	104	90	Y
Nozzle BB #1																102	94	90	Y
Nozzle BB #1 Field Duplicate																86	106	88	Y
Nozzle BB #2												65				104	84	92	Y
Nozzle BB #2 Field Duplicate						845		2890			2920	14				102	94	96	Y
Nozzle BB #2 Field Duplicate RERUN	17700				77200	8450		2850		43400						100	80	96	Y
Nozzle C																102	100	92	Y
Nozzle C Field Duplicate										210	1600	260		9530		98	206	38	N
Nozzle C Field Duplicate RERUN	(SEE 1 BELOW)							44700	42400	2440				9580		100	166	44	N
Nozzle C Field Blank																100	84	92	Y
9/5/84																			
Nozzle BA	7490				128,500	9180		1290					1			108	52	102	N
Nozzle BA Dilution	4340				137,200	4400				9920						104	96	104	Y
Nozzle BB					1804			1540	1573							108	96	96	Y
Nozzle BB Field Blank					173											102	100	104	Y
Nozzle C		446,270	2970	4136	2838					4620	176,405					92	14	98	N
Nozzle C Dilution		283,000	3260	3400	2900			96320	230,400	4340		(SEE 1 BELOW)				102	88	66	N
Nozzle C Field Blank																102	94	80	Y

COMPLETENESS = 19/28 = 68%

NOTES:
1. Sample extracts were diluted, prior to analysis, five times or more. Surrogate recoveries were therefore out of acceptable ranges.
2. Data expressed in mg/kg.
3. All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.

of the GC column revealed sporadic outliers, according to EPA review of these laboratory data. On this basis, quality assurance review suggested strongly that the analytical results presented here are biased low by an amount which cannot be reliably quantitated.

If these data are used for qualitative purposes, some tentative trends or conclusions may be supportable:

- Some of the liquid waste incinerated appeared to contain detectable quantities of benzene ring compounds such as ethylbenzene, styrene, toluene, and xylenes.
- Chlorinated compounds were detected primarily on the third sampling day; however, these findings were largely affected by the surrogate recovery problems highlighted above.
- Of the chlorinated ring compounds, only monochlorobenzene was detected.

A listing of tentatively identified compounds and their concentrations are presented in Table D-11. These data are included for information only, as no support can be offered for their accuracy. Hexamethylcyclotrisiloxane was found in nearly all of the samples and thus appeared to be a laboratory contaminant.

b. Semi-Volatile Compounds

Table D-12 includes data for all quantitated semi-volatile compounds; several target and benzene ring compounds were detected, and accuracy criteria (80-125% surrogate recovery) were met for 15 of the 29 sample runs shown in the table. Note that problems in surrogate recovery occurred chiefly with the acid surrogates. Therefore, the findings of the following compounds may be supported as the surrogate compounds corresponding to their pH range were recovered within acceptable limits:

Waste Nozzle	Sampling Day	Compounds Detected
BB (first feed)	1	2,4,5-trichlorophenol (A) naphthalene (BN) 2-methylnaphthalene (BN)
BA BA	2	2-methylnaphthalene (BN)
BB (first feed)	2	1,2-dichlorobenzene (BN) 2-methylnaphthalene (BN) anthracene (BN)
C	2	1,2-dichlorobenzene (BN) 2,4,5-trichlorophenol (A) 2,4,6-trichlorophenol (A) naphthalene (BN) anthracene (BN) fluorene (BN).

TABLE D-11
LIQUID WASTE INPUTS - TENTATIVELY IDENTIFIED VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, and 9/5/84

	Hexamethylcyclo-trisiloxane	Diethoxydimethyl-silane	2-methylbutane	Cyclohexane	Methoxytrimethyl-silane	2,3-dimethylbutane	(2-methoxyethyl)-trimethylsilane	Ethyl acetate	Ethanol	Propylbenzene	Sulfinylbismethane	1-methylethylbenzene	4-ethenylcyclo-hexane
REAGENT BLANK 1	0.2	821											
REAGENT BLANK 2	0.2	18816											
8/28/84													
Nozzle BA													
Nozzle BA Dilution	0.2		14884	20464	17452	13825							
Nozzle BB #1	0.2	90079					4128						
Nozzle BB #2		7655						1911					
Nozzle BB #2 Dilution													
Nozzle C	0.2	4637							127,972				
Nozzle C RERUN	0.2								127,705				
Field Blank	0.2												
8/30/84													
Nozzle BA	0.2	5375								1071			
Nozzle BA Field Blank	0.2	4219											
Nozzle BB #1	0.2	2333											
Nozzle BB #1 Field Duplicate	0.2	4212											
Nozzle BB #2	0.2	9375									35625		
Nozzle BB #2 Field Duplicate	0.2												
Nozzle BB #2 Field Duplicate RERUN	0.2	27216		7561		10684				1717			
Nozzle C	0.2	6250											
Nozzle C Field Duplicate		2649											
Nozzle C Field Duplicate RERUN	0.2	55492											
Nozzle C Field Blank		1389								4197		869	
9/5/84													
Nozzle BA	0.2	29451	34518	28752									
Nozzle BA Dilution		31960	28446	8630	66477								
Nozzle BB	0.2	12222											
Nozzle BB Field Blank	0.2	2310											
Nozzle C										20086			235,000
Nozzle C Dilution		23333											
Nozzle C Field Blank	0.2	2330											

NOTE: Data expressed in mg/kg.

TABLE D-12
QUANTITATED SEMI-VOLATILE COMPOUNDS - LIQUID WASTE INPUTS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

	TARGET COMPOUNDS				BENZENE RING COMPOUNDS				OTHER	ACCURACY (% SURROGATE RECOVERY)						ACCEPTABLE[1]		
										Base-Neutrals			Acids					
	1,2-dichlorobenzene	Phenol	2,4,5-trichlorophenol	2,4,6-trichlorophenol	Naphthalene	2-methylnaphthalene	Anthracene	Fluorene	Diethylphthalate	Nitrobenzene-D5	2-fluorobiphenyl	Terphenyl - D14	Phenol - D6	2-fluorophenol	2,4,6-tribromophenol	Acid Surrogates	Base-Neutrals	Overall[2]
REAGENT BLANK 1																		
REAGENT BLANK 2																		
8/28/84																		
Nozzle BA										44	60	20	10	8	0	N	Y	N
Nozzle BA, 5X Dilution										25	120	40	15	0	0	N	Y	N
Nozzle BA, 20X Dilution										40	40	60	0	0	0	N	Y	N
Nozzle BB #1			4690		144	77				110	122	52	88	100	92	Y	Y	Y
Nozzle BB #1, 10X Dilution			1900						10500	40	30	40	10	30	0	N	Y	N
Nozzle BB #1, 20X Dilution									60800	20	20	20	20	40	0	N	Y	N
Nozzle BB #2										70	70	20	26	42	20	Y	Y	Y
Nozzle BB #2, 10X Dilution										20	30	20	10	30	0	N	Y	N
Nozzle C										78	84	28	40	32	60	Y	Y	Y
Field Blank (Nozzles BA & BB)										80	88	28	42	86	40	Y	Y	Y
8/30/84																		
Nozzle BA						33			1390	40	40	55	25	40	25	Y	Y	Y
Nozzle BA Field Blank				110					1130	50	60	52	70	94	62	Y	Y	Y
Nozzle BB #1										68	80	32	46	54	54	Y	Y	Y
Nozzle BB #1 Field Duplicate	1406			8320	680	2320		190		132	28	381	16	72	32	N	N	N
Nozzle BB #1 Field Duplicate, 5X Dilution	1570			5570		1950	560			65	30	80	20	30	15	N	Y	N
Nozzle BB #2									5930	60	64	28	44	60	58	Y	Y	Y
Nozzle BB #2 Field Duplicate									110	76	212	178	8	0	0	N	N	N
Nozzle BB #2 Field Duplicate, 10X Dilution										10	30	20	0	0	0	N	N	N
Nozzle C	1240		4450	4490	345		40	145		58	70	62	38	60	70	Y	Y	Y
Nozzle C Field Duplicate					62	27				46	54	22	38	52	38	Y	Y	Y
Nozzle C Field Duplicate, 10X Dilution									270	10	30	30	0	0	0	N	N	N
Nozzle C Field Blank										60	52	38	38	42	40	Y	Y	Y
9/5/84																		
Nozzle BA										52	66	70	2	0	0	N	Y	N
Nozzle BA, 10X Dilution										10	20	20	0	0	0	N	N	N
Nozzle BB										78	82	34	66	50	70	Y	Y	Y
Nozzle BB Field Blank										86	100	118	36	100	60	Y	Y	Y
Nozzle C										68	74	66	46	28	28	Y	Y	Y
Nozzle C, 10X Dilution										30	30	30	20	20	0	N	Y	N
Nozzle C Field Blank										76	58	52	44	64	42	Y	Y	Y
												COMPLETENESS[3], %				52	83	52

NOTES: Data expressed in mg/kg.

[1] All surrogate recoveries within target range (20-180%) established in Quality Assurance Project Plan. N=No, Y=Yes

[2] Based on all surrogate recoveries for both acids and base-neutrals within target range.

[3] By category of surrogates and overall.

[4] Circled data indicate that although accuracy for all surrogates was not acceptable, the recovery of the surrogates corresponding to the pH range of these compounds was within range

In the above summary, "A" in parentheses denotes an acid compound while "BN" denotes a base-neutral compound. The detection of 2,4,6-trichlorophenol, an acid, in the first nozzle BB feed on the second sampling day is not confirmed as the recoveries of all acid surrogates in those samples was not within the acceptable range. Note that diethyl phthalate, a common analytical contaminant, was detected on occasion, and that analytical precision between sample dilutions appeared generally poor.

In Tables D-13, D-14, and D-15 listings of tentatively identified semivolatile compounds are presented sample by sample. In addition to these tentatively identified compounds, a number of peaks labeled "unknown" were listed.

c. Pesticides and Polychlorinated Biphenyls (PCBs)

These data are presented in Table D-16. Most pesticides were detected on the first and second sampling days, with no PCB found in any sample. However, detection limits for the PCBs and for chlordane and toxaphene were in the range of 1 to 10 mg/kg (ppm), much higher than the 5 ppb detection limit specified for this study. Also, as shown in Table D-16, no surrogate recovery data were submitted by the analytical laboratory. Therefore, no judgments can be made concerning the accuracy of these results.

d. PCDD/PCDF

(1) All Homologues

These data, presented with accuracy information in Table D-17 and with detection limit data in Table D-18, show the presence of a wide range of PCDD and PCDF homologues in waste feeds from nozzles BB (first waste fed on the second sampling day) and C. Precision data indicate generally good agreement between the two field duplicate samples obtained on that day. Detection limit goals of 30 ppq for TCDD and TCDF, and 90 ppq for other homologues, were generally met for the latter; however, more frequent problems appeared on the second sampling day, where higher detection limits were common. The completeness criterion of 90%, based upon successful recoveries of all four surrogate compounds, was not met (see Table D-17).

(2) TCDD Isomers

These data are self-explanatory, and are shown in Table D-20, with detection limit data included. Table D-19 is an abridged version of this table, indicating only those isomers which were detected, and rounding the data as appropriate.

2. Low-BTU Liquid Waste (Dike Water)

Dilute wastewaters composed of collected precipitation, condensates from tank farm carbon adsorption system regeneration, and collected runoff from hydroblasting operations in the Dow facility, were incinerated on the first and third sampling days.

TABLE D-13

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

REAGENT BLANK

10.				
11. 541-05-9	HMCTS	ABN	308	70,112
12.				
13.				
14.				
15.				

TABLE D-13 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BA
8/28/84

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. ~~541-05-9~~	~~hexamethylcyclotrisiloxane~~	~~VOA~~		
2.	unknown	ABN	331	
3. 100-41-4	C_8H_{10} - ethylbenzene		351	1,337,410
4. 611-14-3	C_9H_{12} - 1-ethyl-2-methylbenzene		486	1,007,220
5. 620-14-4	C_9H_{12} - 1-ethyl-3-methylbenzene		507	1,046,048
6.	unknown		559	
7.	"		582	
8. 54461-45-1	$C_9H_{11}OCl$ - 4-Chloro-2-(1-methyl ethyl) phenol		648	1,289,685
9.				
10.	unknown		695	
11.			702	
12.			793	
13.			919	
14.	~~[illegible]~~ - unknown		1017	
15.			1098	
16.			1128	
17.			1258	
18.			1567	
19. 54-15-1	$C_{18}H_{14}$ - 1,1':2',1"- terphenyl		1291	1,150,036
20. 92-06-8	C_8H_{14} - 1,1':3',1" - terphenyl		1448	427,605
21.				

TABLE D-13 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BA

8/28/84 LABORATORY RERUN

CAS Number	Compound Name	Fraction	RT or [✓ Scan] Number	Estimated Concentration (ug/l or [✓ ug/kg])
1.	unknown	ABN	223	
2.	"		234	
3. 681-84-5	$C_4H_{12}O_4Si$ - silicic acid, Tetra		261	4,272,520
4.	methylester			
5. 127-18-4	C_2Cl_4 - Tetrachloroethene		287	4,342,120
6. 108-90-7	C_6H_5Cl - chlorobenzene		338	11,035,105
7.	unknown		692	
8.	"		918	
9. 776-76-1	$C_{13}H_{14}Si$ - methyldiphenylsilane		1017	18,699,780
10. 605-39-0	$C_{14}H_{14}$ - 2-ethyl-1,1'-biphenyl		1085	396,288
11.	unknown		1101	
12. 144-79-6	$C_{13}H_{13}ClSi$ - chloromethyldiphenyl		1130	9,781,986
13.	silane			
14. 588-59-0	$C_{14}H_{12}$ - 1,1'-(1,2-ethendiyl)bis-		1170	1,184,490
15.	benzene			
16.	unknown		1213	
17. 84-15-1	$C_{18}H_{14}$ - 1,1':2',1"-terphenyl		1294	1,693,644
18. 92-06-8	$C_{18}H_{14}$ - 1,1':3',1"- Terphenyl		1453	953,650
19.	"		1477	
20.	unknown	↓	1493	
21.				

TABLE D-13 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BA

8/28/84 SECOND LABORATORY RERUN

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. ~~541-05-9~~	~~hexamethylcyclotrisiloxane~~	~~VOA~~		
2. 127-18-4	C_2Cl_4 - tetrachloroethene	ABN	286	5,616,186
3. 108-90-7	C_6H_5Cl - chlorobenzene		335	14,331,174
4.	unknown		340	
5. 100-41-4	C_8H_{10} - ethylbenzene		357	2,647,871
6.	unknown		467	
7. 611-14-3	C_9H_{12} - 1-ethyl-2-methylbenzene		487	1,751,619
8.	unknown		571	
9.	"		580	
10. 54461-05-1	$C_9H_{11}OCl$ - 4-Chloro-2-(1-methyl		644	2,452,347
11.	ethyl) phenol			
12.	unknown		688	
13.			697	
14.			790	
15.	↓		909	
16. 1812-51-7	$C_{14}H_{14}$ - 2-ethyl-1,1'-biphenyl		979	1,113,816
17. 776-76-1	$C_{13}H_{14}Si$ - methyldiphenylsilane		1010	22,390,761
18.	unknown		1094	
19. 144-79-6	$C_{13}H_{13}ClSi$ - chloromethyldiphenyl		1123	16,602,557
20.	silane			
21. 588-59-0	$C_{14}H_{12}$ - 1,1'-(1,2-ethenediyl) bis		1162	1,586,912
22.	benzene			
23. 84-15-1	$C_{18}H_{14}$ - 1,1':2',1"-terphenyl		1288	7,776,799
24. 26140-60-3	$C_{18}H_{14}$ - terphenyl	↓	1469	2,939,694
25.				

TABLE D-13 (cont.)
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BB (1145-1606 EDT)
8/28/84

12. 103-65-1	C_9H_{12} - propyl benzene		478	1,249,834
13.	unknown		565	
14.	↓		801	
15.			813	
16. 112-50-5	$C_8H_{18}O_4$ - 2-\2-(2-ethoxy)ethoxy		838	994,430
17.	ethanol			
18. 92-52-4	$C_{12}H_{10}$ - biphenyl		914	3,552,668
19. 101-84-8	$C_{12}H_{10}O$ - 1,1'-oxybisbenzene		935	20,945,053
20.	unknown		986	
21.	"		1069	
22. 100-42-5	C_8H_8 - ethenylbenzene		1198	5,235,434
23.	unknown		1579	
24.	↓		1615	
25.		↓	1685	

TABLE D-13 (cont.)
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BB (1145-1606 EDT) LABORATORY RERUN

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. 541-05-9	hexamethylcyclotrisiloxane	ABN	314	621,000
2. 67-68-5	C_2H_6OS - sulfinylbismethane		323	4,600,857
3. 100-41-4	C_8H_{10} - ethylbenzene		358	14,545,537
4. 108-38-3	C_8H_{10} - 1,3-dimethylbenzene		368	1,305,087
5.	unknown		382	
6. 694-87-1	C_8H_8 - bicyclo(4.2.0)octa,1,3,5-		397	6,074,000
7.	triene			
8. 98-82-8	C_9H_{12} - (1-methylethyl) benzene		441	1,026,544
9. 122-99-6	$C_8H_{10}O_2$ - 2-phenoxyethanol		782	31,378,000
10. 4169-04-4	$C_9H_{12}O_2$ - 2-phenoxy-1-propanol		799	5,849,530
11.	unknown		1025	
12.	C_8H_8 - styrene		1159	598,930
13. 92-69-3	$C_{12}H_{10}O$ - 1,1'-biphenyl-4-ol		1185	586,300
14.	unknown	↓	1199	
15.				

TABLE D-13 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BB (1145-1606 EDT) - 8/28/84 SECOND LABORATORY RERUN

	CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1.	~~541-05-9~~	~~hexamethylcyclotrisiloxane~~	~~VOA~~		
2.	67-68-5	C_2H_6OS - sulfinylbismethane	ABN	316	6,271,900
3.	584-03-2	$C_4H_{10}O_2$ - 1,2-butanediol		340	3,979,208
4.	100-41-4	C_8H_{10} - ethylbenzene		356	18,635,353
5.		unknown		379	
6.	694-87-1	C_8H_8 - bicyclo(4.2.0)octa 1,3,5		395	7,961,452
7.		triene			
8.	111-76-2	$C_6H_{14}O_2$ - 2-butoxyethanol		418	3,973,380
9.	122-99-6	$C_8H_{10}O_2$ - 2-phenoxyethanol		769	66,845,824
10.	4169-04-4	$C_9H_{12}O_2$ - 2-phenoxy-1-propanol		790	10,797,507
11.	92-52-4	$C_{12}H_{10}$ - 1,1'-biphenyl		906	3,281,094
12.	101-84-8	$C_{12}H_{10}O$ - 1,1'-oxybisbenzene		926	9,880,923
13.		unknown		1193	
14.					

TABLE D-13 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BB (1606-2020 EDT)

8/28/84

10.		unknown	ABN	391	
11.				736	
12.				743	
13.				822	
14.				840	
15.				883	
16.				977	
17.				987	
18.				1007	
19.				1016	
20.				1030	
21.				1050	
22.				1068	
23.				1132	
24.				1565	
25.					
26.					

TABLE D-13 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BB (1606-2020 EDT)
8/28/84 LABORATORY RERUN

CAS Number	Compound Name	Fraction	RT or [✓ Scan] Number	Estimated Concentration (ug/l or [✓ ug/kg])
1. 541-05-9	hexemethylcyclotrisiloxane	ABN	314	611,955
2.	unknown	↓	396	449,840
3.	↓		881	
4.			1044	
5.			1067	
6.			1093	
7.				

TABLE D-13 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE C
8/28/84

10.				
11. 108-88-3	C_7H_8 - methylbenzene	ABN	216	2,512,400
12.	unknown	↓	967	
13. 719-22-2	$C_{14}H_{20}O_2$ - 2,6-bis(1,1-dimethyl-		987	866,883
14.	ethyl)-2-5-cyclohexadiene			
15.	-1,4-dione			
16.	unknown		1117	
17. 1460-02-2	$C_{18}H_{30}$ - 1,3,5-Tris (1,1-dimethylethyl)		1145	177,600
18.	benzene			
19.	unknown		1268	
20.	↓		1487	
21.			1829	
22.			1896	
23.			2026	
24.			2885	
25.			2893	
26.			2937	
27.				

TABLE D-13 (cont.)
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE FIELD BLANK - 8/28/84

10.				
11.	none found	ABN		
12.				
13.				
14.				

TABLE D-14
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BA - 8/30/84

10.				
11. 67-68-5	C_2H_6OS - sulfinylbismethane	ABN	339	4,251,000
12. 108-38-3	C_8H_{10} - 1,3-dimethylbenzene	↓	365	20,019,000
13.	unknown		372	
14.	↓		392	
15.			406	
16. 111-76-2	$C_6H_{14}O_2$ - 2-butoxyethanol		428	2,291,700
17. 98-82-8	C_9H_{12} - (1-methylethyl) benzene		445	2,555,770
18. 103-65-1	C_9H_{12} - propyl benzene		480	1,737,245
19.	unknown		643	
20.	↓		781	
21.			939	
22.			1026	
23. 1081-75-0	$C_{15}H_{16}$ - 1,1'-(1,3-propanediyl) bis-benzene		1128	730,180
24.				
25. 100-42-5	C_8H_8 - ethenylbenzene		1200	1,328,920
26.	unknown		1227	
27.	↓		1618	
28.			1680	
29.			1690	
30.				

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BB (1000-1415 EDT) FIELD DUPLICATE SAMPLE

8/30/84

11.	1120-21-4	$C_{11}H_{24}$ - undecane	ABN	649	3,296,037
12.	934-74-7	$C_{10}H_{14}$ - 1-ethyl-3,5-dimethyl-		700	2,101,512
13.		benzene			
14.	112-40-3	$C_{12}H_{26}$ - dodecane		749	4,563,513
15.	112-35-6	$C_7H_{16}O_4$ - 2-[2-(2-methoxyethoxy)		787	10,490,388
16.		ethoxy ethanol			
17.	2809-64-5	$C_{11}H_{14}$ - Tetrahydro-5-methyl		808	1,463,038
18.		naphthalene			
19.	17301-33-6	$C_{13}H_{28}$ - 4,8-dimethylundecane		841	8,953,388
20.	90-12-0	$C_{11}H_{10}$ - 1-methylnaphthalene		852	2,328,550
21.	933-75-5	$C_6H_3OCl_3$ - 2,3,6-Trichlorophenol		902	3,753,750
22	629-59-4	$C_{14}H_{30}$ - Tetradecane		928	9,467,662
23.	582-16-1	$C_{12}H_{12}$ - 2,7-dimethylnaphthalene		950	5,366,388
24.	629-62-9	$C_{15}H_{32}$ - pentadecane		1011	4,790,075
25.		~~[illegible]~~ - unknown		1061	
26	112-40-3	$C_{12}H_{26}$ - dodecane		1160	5,113,587
27.		unknown		1186	
28.		"		1233	
29	54352-74-3	$C_{14}H_{14}O$ - ethylphenoxybenzene		1292	4,086,637
30.		~~[illegible]~~ unknown		1365	
		"		1505	
		"		1544	
		"	↓	1557	

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BB (1000-1415 EDT) FIELD DUPLICATE SAMPLE LABORATORY RERUN

8/30/84

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. 526-73-8	C_9H_{12} - 1,2,3-trimethylbenzene	ABN	524	7,876
2. 112-40-3	$C_{12}H_{26}$ - dodecane		744	3,526,292
3. 112-35-6	$C_7H_{16}O_4$ - 2-\2-(2-methoxyethoxy)		764	1,892,111
4.	ethoxy ethanol			
5. 112-35-6	$C_7H_{16}O_4$ - ~~[illegible]~~ "		772	5,929,720
6.	unknown		1011	
7. 92-69-3	$C_{12}H_{10}O$ - 1,1'-biphenyl-4-ol		1164	636,163
8.	unknown		1193	
9.			1209	
10.	↓		1231	
11. 54852-74-3	$C_{14}H_{14}O$ - ethylphenoxybenzene		1279	20,489,893
12.	unknown		1306	
13. 6738-04-1	$C_{18}H_{14}O$ - 2-phenoxy-1,1'-biphenyl		1356	69,712,565
14. 3933094-6	$C_{18}H_{14}O$ - 4-phenoxy-1,1'-biphenyl		1475	12,307,486
15. 2432-11-3	$C_{18}H_{14}O$ - 1,1':3',1" terphenyl-2-ol		1497	19,347,100
16.	"	↓	1545	142,226,700
17.				
18.				

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BA FIELD BLANK - 8/30/84

11.	unknown	ABN	185	
12.	"		347	
13. 112-35-6	$C_7H_{16}O_4$ - 2-\2-(2-methoxy-		769	964,960
14.	ethoxy) ethoxy ethanol			
15. 122-99-6	$C_8H_{10}O_2$ - 2-phenoxyethanol		774	413,166
16. 112-35-6	$C_7H_{16}O_4$ - 2-\2-(2-methoxyethoxy)		1008	566,841
17.	ethoxy methanol			
18. 92-69-3	$C_{12}H_{10}O$ - 1,1'-biphenyl-4-ol	↓	1179	692,840
19.				
20.				
21.				

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BB (1000-1415 EDT) - 8/30/84

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. 541-05-9	hexamethylcyclotrisiloxane	ABN	309	103,186
2.	unknown	↓	593	
3. 1541-20-4	$C_{12}H_{18}$ - bi-2-cyclohexen-1-yl		606	52,800
4.				
5.				
6.				
7.				

TABLE D-14 (cont.)
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BB (1415-1700 EDT) - 8/30/84

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. 541-05-9	hexamethylcyclotrisiloxane	ABN	310	140,364
2.	unknown	↓	342	
3.			346	
4. 112-35-6	$C_7H_{16}O_4$ - 2-[2-(2-methoxyethoxy)		769	582,765
5.	ethoxy ethanol			
6. 122-99-6	$C_8H_{10}O_2$ - 2-phenoxyethanol		775	265,970
7. 4169-04-4	$C_9H_{12}O_2$ - 2-phenoxy-1-propanol		796	37,130
8. 88-99-3	$C_8H_6O_4$ - 1,2-benzenedicarboxylic		859	115,375
9.	acid			
10. 112-35-6	$C_7H_{16}O_4$ - 2-[2-(2-methoxyethoxy)	↓	1011	421,930
11.	ethoxy ethanol			
12.				

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE C - 8/30/84

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. 1120-21-4	$C_{11}H_{24}$ - undecane	ABN	648	186,340
2. 934-74-7	$C_{10}H_{14}$ - 1-ethyl-3,5-dimethyl-		700	1,349,060
3.	benzene			
4. 112-40-3	$C_{12}H_{26}$ - dodecane		749	2,812,895
5. 112-35-6	$C_7H_{16}O_4$ - 2-\2-(2-methoxyethoxy)		795	2,874,400
6.	ethoxy ethanol			
7.	unknown		842	
8. 90-12-0	$C_{11}H_{10}$ - 1-methylnaphthalene		853	1,476,094
9.	unknown		908	
10. 629-59-4	$C_{14}H_{30}$ - Tetradecane		928	4,081,220
11. 582-16-1	$C_{12}H_{12}$ - 2,7-dimethylnaphthalene		935	1,191,737
12. 573-98-8	$C_{12}H_{12}$ - 1,2-dimethylnaphthalene		948	3,035,520
13. 575-41-7	$C_{12}H_{12}$ - 1,3- " "		963	749,926
14.	unknown		978	
15.	↓		987	
16.			1009	
17. 90-43-7	$C_{12}H_{10}O$ - 1,1'-biphenyl-2-ol		1030	1,716,077
18. 829-26-5	$C_{13}H_{14}$ - 2,3,6-Trimethylnaphthalene		1037	441,374
19. 2245-38-7	$C_{13}H_{14}$ - 1,6,7- " "		1049	1,953,200
20.	unknown		1057	
21. 3031-08-1	$C_{13}H_{14}$ → Trimethylnaphthalene	↓	1062	930,994
22.	\(1,3,6)			
23.				

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE C - 8/30/84 FIELD BLANK

7.	unknown	ABN	593	
8.				
9.				
10.				
11.				
12.				

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE C - 8/30/84 FIELD DUPLICATE SAMPLE

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. ~~541-05-9~~	~~hexamethylcyclotrisiloxane~~	~~VOA~~		
2.	unknown		483	
3. 1112-39-6	$C_4H_{12}O_2Si$ - dimethoxydimethyl-		515	2,649,050
4.	silane			
5.	unknown		730	
6.	↓		858	
7.			884	
8. 103-65-1	C_9H_{12} - propylbenzene		938	4,196,510
9.	unknown		969	
10. 25013-15-4	C_9H_{10} - 1-methylethylbenzene	↓	983	868,984
11.	unknown	ABN	360	
12. 108-38-3	C_8H_{10} - 1,3-dimethylbenzene		366	1,197,205
13.	unknown		380	
14.	↓		393	
15.			404	
16. 98-82-8	C_9H_{12} - 1-methylethylbenzene		441	1,714,350
17. 103-65-1	C_9H_{12} - propylbenzene		478	1,111,011
18. 3188-00-9	$C_5H_8O_2$ - dihydro-5-methyl-		642	1,313,670
19.	3(2H) furanone			
20.	unknown		783	
21. 4169-04-4	$C_9H_{12}O_2$ - 2-phenoxy-1-propanol		801	2,102,061
22.	unknown		916	
23.			940	
24.			980	
25.	↓		1024	
26. 1081-75-0	$C_{15}H_{16}$ - 1,1'-(1,3-propanediyl)		1125	545,109
7.	bis benzene			
8. 92-69-3	$C_{12}H_{10}O$ - 1,1'-biphenyl-4-ol		1184	651,947
9. 57-10-3	$C_{16}H_{32}O_2$ - hexadecanoic acid		1337	608,496
0.	unknown		1616	
	〃	↓	1688	

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE C - 8/30/84 FIELD DUPLICATE SAMPLE LABORATORY RERUN

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1. 108-87-2	C_7H_{14} - methylcyclohexane	ABN	183	1,562,700
2. 108-88-3	C_7H_8 - methylbenzene		229	2,118,480
3. 67-68-5	C_2H_6OS - sulfinylbismethane		323	2,841,125
4. 108-38-3	C_8H_{10} - 1,3-dimethylbenzene		359	27,596,480
5. 106-42-3	C_8H_{10} - 1,4- "		368	1,829,170
6.	unknown		384	
7. 629-20-9	C_8H_8 - 1,3,5,7-cyclooctatetraene		400	33,301,400
8. 111-76-2	$C_6H_{14}O_2$ - 2-butoxyethanol		423	1,817,024
9. 98-82-8	C_9H_{12} - 1-methylethylbenzene		441	2,584,080
10. 122-99-6	$C_8H_{10}O_2$ - 2-phenoxyethanol		778	14,566,897
11. 4169-04-4	$C_9H_{12}O_2$ - 2-phenoxy-1-propanol		797	2,552,806
12. 92-52-4	$C_{12}H_{10}$ - 1,1'-biphenyl		913	15,155,310
13. 101-84-8	$C_{12}H_{10}O$ - 1,1'-oxybisbenzene		934	35,799,221
14.	unknown	↓	1197	
15.				

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BB (1415-1700 EDT) FIELD DUPLICATE SAMPLE

8/30/84

11.	unknown	ABN	344	
12.			398	
13.	↓		471	
14. 611-14-3	C_9H_{12} - 1-ethyl-2-methylbenzene		492	1,642,857
15. 95-63-6	C_9H_{12} - 1,2,4-trimethylbenzene		513	2,357,142
16.	unknown		517	
17.			564	
18.	↓		581	
19. 5446-05-1	$C_9H_{11}OCl$ - 4-chloro-2-(1-methyl		651	2,571,428
20.	ethyl) phenol			
21.	unknown		699	
22.			709	
23.			739	
24.			769	
25.			796	
26.			927	
27.			1023	
28.	↓		1298	
29. 92-06-8	$C_{18}H_{14}$ - 1,1':3',1"-terphenyl		1481	2,928,571
30.	unknown	↓	1576	

TABLE D-14 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE BB (1415-1700 EDT) FIELD DUPLICATE SAMPLE LABORATORY RERUN

8/30/84

10.					
11.		unknown	ABN	233	
12.	681-84-5	$C_4H_{12}O_4Si$ - silicic acid tetramethyl		261	8,366,867
13.		ester		[illegible]	
14.		unknown		278	
15.	127-18-4	C_2Cl_4 - tetrachloroethene		287	5,278,348
16.	1186-53-4	C_9H_{20} - 2,2,3,4-tetramethylpentane		298	1,432,650
17.		unknown		317	
18.	108-90-7	C_6H_5Cl - chlorobenzene		337	14,440,300
19.		unknown		341	
20.	100-41-4	C_8H_{10} - ethylbenzene		359	3,098,514
21.	108-38-3	C_8H_{10} - 1,3-dimethylbenzene		369	2,019,550
22.		unknown		375	
23.		"		395	
24.	611-14-3	C_9H_{12} - 1-ethyl-2-methylbenzene		488	1,873,390
25.	611-14-3	C_9H_{12} - " "		509	1,781,300
26.	[illegible]-55-1	$C_9H_{11}OCl$ - 4-chloro-2-(1-methyl		646	1,072,580
27.		ethyl) phenol			
28.		unknown		689	
29.		unknown aromatic		914	
30.	1812-51-7	$C_{14}H_{14}$ - 2-ethyl-biphenyl		981	898,230
31.	776-76-1	$C_{13}H_{14}Si$ - methyldiphenylsilane		1013	18,418,960
32.	144-79-6	$C_{13}H_{13}ClSi$ - chloromethyldiphenyl- silane		1124	4,881,264
33.	84-15-1	$C_{18}H_{14}$ - 1,1':2',1"- terphenyl		1289	1,560,000
34.	92-06-8	$C_{18}H_{14}$ - 1,1':3',1"- terphenyl	↓	1448	884,913

TABLE D-15
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BA
9/5/84

11.	unknown	ABN	338	
12. 100-41-4	C_8H_{10} - ethylbenzene		358	1,866,185
13.	unknown		379	
14.	"		437	
15. 103-65-1	C_9H_{12} - propylbenzene		479	841,741
16. 611-14-3	C_9H_{12} - 1-ethyl-2-methyl		490	1,253,012
17.	benzene			
18.	unknown		495	
19.			512	
20.			538	
21.			577	
22.			589	
23 54461-03-1	$C_9H_{11}OCl$ - 4-chloro-2-(1-methyl		650	1,662,151
24.	ethyl) phenol			
25.	unknown		697	
26.	"		920	
27. 776-76-1	$C_{13}H_{14}Si$ - methyldiphenylsilane		1017	6,218,166
28.	unknown		1098	
29.	"		1131	
30.	"		1262	
31	unknown		1294	
30. 92-06-8	$C_{18}H_{14}$ - 1,1':3',1''-Terphenyl		1477	862,400
33.	unknown		1573 --	

TABLE D-15 (cont.)
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BA LABORATORY RERUN - 9/5/84

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1.	unknown	ABN	223	
2.	"		227	
3. 108-88-3	C_7H_8 - methylbenzene		232	150,869,980
4. 127-18-4	C_2Cl_4 - Tetrachloroethene		285	13,266,160
5. 1186-53-4	C_9H_{20} - 2,2,3,4-Tetramethyl-		296	1,228,790
6.	pentane			
7. 108-90-7	C_6H_5Cl - chlorobenzene		335	7,416,630
8.	unknown		397	
9.			509	
10.			668	
11. 776-76-1	$C_{13}H_{14}Si$ - methyldiphenylsilane		1010	4,920,597
12.				
13.				

TABLE D-15 (cont.)
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BB FIELD BLANK
9/5/84

10.				
11.	none found	ABN		
12.				
13.				
14.				
15.				
16.				

TABLE D-15 (cont.)
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE BB
9/5/84

11. 109-02-4	$C_5H_{11}ON$ - 4-methylmorpoline	ABN	250	560,600
12.	unknown		345	
13. 100-41-4	C_8H_{10} - ethylbenzene		356	1,163,500
14. 100-42-5	C_8H_8 - ethenylbenzene		397	1,672,200
15.	unknown		576	
16.	"		649	
17. 598-94-71	$C_3H_8ON_2$ - N,N-dimethylurea		663	968,500
18.	unknown		710	
19. 122-98-6	$C_8H_{10}O_2$ - 2-phenoxyphenol		783	983,417
20.	unknown		801	
21. 101-84-8	$C_{12}H_{10}O$ - 1,1'-oxybis benzene		935	2,332,118
22.				

TABLE D-15 (cont.)
LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
LIQUID WASTE NOZZLE C
9/5/84

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1.	unknown	ABN	229	
2.	"		371	
3.			447	
4.			468	
5.			476	
6.			485	
7.			496	
8.			502	

TABLE D-15 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE C LABORATORY RERUN - 9/5/84

10.					
11.	100-40-3	C_8H_{12} - 4-ethenylcyclohexene	ABN	323	54,219,890
12.	10.-42-3	C_8H_{10} - 1,4-dimethylbenzene	↓	361	62,774,545
13.	100-41-4	C_8H_{10} - ethylbenzene		369	2,641,023
14.		unknown		408	
15.	41898-89-9	C_9H_{12} - 2,4-dimethyl-2,3-hepta-		444	25,595,417
16.		diene-5-yne			
17.	300-57-2	C_9H_{10} - 2-propenylbenzene		468	4,463,125
18.	103-65-1	C_9H_{12} - propylbenzene		478	9,963,116
19.	100-52-7	C_7H_6O - benzaldehyde		484	4,127,603
20.	4994-16-5	$C_{12}H_{14}$ - 3-cyclohexen-1-yl-		866	1,330,570
21.		benzene			
22.					

TABLE D-15 (cont.)

LIQUID WASTE INPUTS - TENTATIVELY-IDENTIFIED SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

LIQUID WASTE NOZZLE C FIELD BLANK
9/5/84

11.		none found	ABN		
12.					
13.					
14.					
15.					
16.					

TABLE D-16
LIQUID WASTE INPUTS - QUANTITATED PESTICIDE/PCB COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, and 9/5/84

	PESTICIDES											PCB (AROCLORS)							ACCURACY				(% SURROGATE RECOVERY)							
	Aldrin	Alpha - BHC	Beta - BHC	Gamma - BHC (Lindane)	Chlordane	4,4'-DDD	4,4'-DDT	Dieldrin	Endosulfan II	Heptachlor	Toxaphene	1016	1221	1232	1242	1248	1254	1260												
REAGENT BLANK 1																														
REAGENT BLANK 2																														
8/28/84																							(ACCURACY DATA NOT							
Nozzle BA				0.3																										
Nozzle BB #1				0.3																			SUBMITTED BY							
Nozzle BB #2										0.1																				
Nozzle C																							ANALYTICAL							
Nozzles BA & BB Field Blank																														
8/30/84																							LABORATORY.)							
Nozzle BA	1.4	7.5						2.5																						
Nozzle BA Field Blank																														
Nozzle BB #1																														
Nozzle BB #1 Field Duplicate						0.4	0.6		0.4																					
Nozzle BB #2																														
Nozzle BB #2 Field Duplicate				0.8																										
Nozzle C																														
Nozzle C Field Duplicate	3.1	11.7				1.2		3.1	1.4	1.2																				
Nozzle C Field Blank																														
9/5/84																														
Nozzle BA			0.2	0.1																										
Nozzle BB																														
Nozzle BB Field Blank																														
Nozzle C							0.3																							
Nozzle C Field Blank																														

NOTE: Data expressed in mg/kg. Where data are not stated, compound was not detected.

TABLE D-17
LIQUID WASTE INPUTS - QUANTITATED PCDD/PCDF[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, and 9/5/84

													ACCURACY (% RECOVERY)			
	2378 - TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378 - TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	$^{13}C_{12}$-2378-TCDD	$^{37}Cl_4$-2378-TCDD	$^{13}C_{12}$ - OCDD	$^{37}Cl_4$-2378-TCDF
REAGENT BLANK 1																
REAGENT BLANK 2																
8/28/84																
Nozzle BA													35	119	100	44
Nozzle BA Field Blank													100	90	89	61
Nozzle BB #1		5.8	11.8	1.2	2.8	22.0		9.2	0.8			1.2	38	118	100	39
Nozzle BB #2		0.5						0.4					88	112	100	75
Nozzle C													95	105	100	81
8/30/84																
Nozzle BA													88	96	100	84
Nozzle BA Field Blank													100	91	34	82
Nozzle BB #1		33.0	6.3	0.9	3.0	11.5	0.3	37.0	1.8	0.7	0.6	0.6	100	90	87	75
Nozzle BB #1 Field Duplicate		30.7	4.9	0.4	2.6	12.1	1.4	32.3	5.3				100	113	92	85
Nozzle BB #2													77	88	83	75
Nozzle BB #2 Field Duplicate													57	94	100	60
Nozzle C		60.3	3.5	2.6	3.8	19.8		36.6	1.5	3.5	8.1	7.4	75	98	53	100
Nozzle C Field Duplicate		21.8	6.1	4.2	5.7	19.8	2.1	18.0	4.3	7.1	8.2	7.7	97	97	23	100
Nozzle C Field Blank													100	93	49	57
9/5/84																
Nozzle BA													100	93	75	50
Nozzle BB		5.9	0.8			1.2	0.2	6.5	0.2				100	91	53	90
Nozzle BA Field Blank		(SAMPLE ANALYSIS NOT RETURNED FROM LABORATORY)														
Nozzle C		0.8						0.2					99	92	40	100
Nozzle C Field Blank													75	99	64	56
						COMPLETENESS BY SURROGATE							84%	95%	74%	84%

NOTES: 1. Data expressed in ng/g.
2. All surrogate recoveries within target ranges of 50-150%.

TABLE D-18
LIQUID WASTE INPUTS - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	TOTAL TCDD	TOTAL PeCDD	TOTAL HxCDD	TOTAL HpCDD	OCDD	2378-TCDF	TOTAL TCDF	TOTAL PeCDF	TOTAL HxCDF	TOTAL HpCDF	OCDF
8/28/84												
Nozzle BA	ND(0.0066)	ND(0.0419)	ND(0.295)	ND(0.399)	ND(0.321)	ND(0.204)	ND(0.0076)	ND(0.0436)	ND(0.226)	ND(0.0992)	ND(0.289)	ND(0.0532)
Nozzle BA Field Blank	ND(0.0199)	ND(0.0322)	ND(0.183)	ND(0.107)	ND(0.557)	ND(0.772)	ND(0.0183)	ND(0.0788)	ND(0.128)	ND(0.0531)	ND(0.317)	ND(0.0429)
Nozzle BB #1	ND(0.110)	5.79	11.8	1.19	2.79	22.0	ND(0.166)	9.15	0.845	ND(0.108)	ND(0.242)	1.24
Nozzle BB #2	ND(0.0128)	0.548	ND(0.371)	ND(0.0782)	ND(0.265)	ND(0.828)	ND(0.0128)	0.373	ND(0.0420)	ND(0.0199)	ND(0.0831)	ND(0.333)
Nozzle C	ND(0.0156)	ND(0.0173)	ND(0.0398)	ND(0.0294)	ND(0.126)	ND(0.339)	ND(0.0095)	ND(0.0289)	ND(0.0164)	ND(0.0346)	ND(0.0473)	ND(0.329)
8/30/84												
Nozzle BA	ND(0.0035)	ND(0.0098)	ND(0.0289)	ND(0.0757)	ND(0.122)	ND(0.183)	ND(0.0065)	ND(0.0131)	ND(0.0436)	ND(0.0391)	ND(0.0599)	ND(0.148)
Nozzle BA Field Blank	ND(0.0117)	ND(0.0044)	ND(0.0188)	ND(0.0094)	ND(0.0389)	ND(0.0551)	ND(0.0030)	ND(0.0045)	ND(0.0102)	ND(0.0087)	ND(0.0313)	ND(0.0528)
Nozzle BB #1	ND(0.0416)	33.0	6.27	0.895	3.00	11.5	0.284	37.0	1.78	0.749	0.593	0.625
Nozzle BB #1 Field Duplicate	ND(0.0603)	30.7	4.85	0.375	2.64	12.1	1.43	32.3	5.33	ND(0.0585)	ND(0.0873)	ND(1.41)
Nozzle BB #2	ND(0.0349)	ND(0.0208)	ND(0.0319)	ND(0.135)	ND(0.266)	ND(0.314)	ND(0.0268)	ND(0.0126)	ND(0.0515)	ND(0.0867)	ND(0.0232)	ND(0.260)
Nozzle BB #2 Field Duplicate	ND(0.0122)	0.716	ND(0.0661)	ND(0.309)	ND(0.0567)	ND(0.0522)	ND(0.0240)	0.819	ND(0.0134)	ND(0.0391)	ND(0.0765)	ND(0.0480)
Nozzle C	ND(0.152)	60.3	3.45	2.61	3.80	19.8	ND(0.126)	36.6	1.51	3.51	8.07	7.43
Nozzle C Field Blank	ND(0.0396)	ND(0.0394)	ND(0.109)	ND(0.0693)	ND(0.211)	ND(0.0715)	ND(0.0392)	ND(0.0614)	ND(0.0603)	ND(0.166)	ND(0.269)	ND(0.224)
Nozzle C Field Duplicate	ND(0.108)	21.8	6.13	4.24	5.69	19.8	2.10	18.0	4.32	7.13	8.16	7.68
9/5/84												
Nozzle BA	ND(0.0094)	ND(0.0013)	ND(0.0084)	ND(0.0046)	ND(0.0107)	ND(0.0158)	ND(0.0037)	ND(0.0037)	ND(0.0117)	ND(0.0053)	ND(0.0065)	ND(0.0124)
Nozzle BA Field Blank												
Nozzle BB	ND(0.0244)	5.88	0.808	ND(0.0892)	ND(0.132)	1.21	0.237	6.48	0.178	ND(0.0639)	ND(0.0674)	ND(0.184)
Nozzle C	ND(0.0796)	0.835	ND(0.235)	ND(0.217)	ND(0.570)	ND(0.723)	ND(0.0574)	0.181	ND(0.116)	ND(0.153)	ND(0.476)	ND(0.450)
Nozzle C Field Blank	ND(0.0096)	ND(0.0027)	ND(0.0041)	ND(0.0031)	ND(0.0148)	ND(0.0207)	ND(0.0038)	ND(0.0046)	ND(0.0028)	ND(0.0037)	ND(0.0115)	ND(0.0153)

NOTES: Data expressed in ng/g.
Accuracy (surrogate recovery) data shown in Table D-17.

TABLE D-19
LIQUID WASTE INPUTS - TCDD ISOMER ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279
8/28/84													
Nozzle BA													
Nozzle BA Field Blank													
Nozzle BB #1	1.2	4.1								0.5			
Nozzle BB #2	0.3	0.3											
Nozzle BB #2 Lab Duplicate													
Nozzle C													
8/30/84													
Nozzle BA													
Nozzle BA Lab Duplicate													
Nozzle BA Field Duplicate													
Nozzle BB #1	21.8	10.3		0.4						0.4			
Nozzle BB #1 Field Duplicate	19.3	11.4											
Nozzle BB #2													
Nozzle BB #2 Field Duplicate													
Nozzle C	39.9	20.4											
Nozzle C Field Duplicate	8.8	10.8		0.4						1.4		0.3	*
Nozzle C Field Blank													
9/5/84													
Nozzle BA													
Nozzle BA Field Blank													
Nozzle BB	4.1	1.8											
Nozzle BB Lab Duplicate	4.2	1.6		0.1						0.1			
Nozzle C	0.6	0.2											
Nozzle C Field Blank													

* GC retention time exceeded; therefore, this isomer could not be quantitated.

NOTE: Data expressed in ng/g.
Blank spaces denote isomer was not detected (see Table D-20).

TABLE D-20
LIQUID WASTE INPUTS - TCDD ISOMERS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
8/28/84															
Nozzle BA	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0066)	ND(0.0419)	ND(0.0419)	ND(0.0419)	ND(0.0419)
Nozzle BA Field Blank	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0199)	ND(0.0322)	ND(0.0322)	ND(0.0322)	ND(0.0322)
Nozzle BB #1	1.19	4.11	ND(0.206)	ND(0.206)	ND(0.206)	ND(0.206)	ND(0.206)	ND(0.206)	ND(0.206)	0.493	ND(0.110)	ND(0.110)	ND(0.206)	ND(0.206)	ND(0.206)
Nozzle BB #2	0.276	0.272	ND(0.0315)	ND(0.0315)	ND(0.0315)	ND(0.0315)	ND(0.0315)	ND(0.0315)	ND(0.0315)	ND(0.0315)	ND(0.0129)	ND(0.0315)	ND(0.0315)	ND(0.0315)	ND(0.0315)
Nozzle BB #2 Lab Duplicate	ND(0.0077)	ND(0.0033)	ND(0.0033)	ND(0.0033)	ND(0.0033)	ND(0.0033)	ND(0.0033)	ND(0.0033)	ND(0.0033)	ND(0.0033)	ND(0.0108)	ND(0.0033)	ND(0.0101)	ND(0.0033)	ND(0.0397)
Nozzle C	ND(0.0173)	ND(0.0173)	ND(0.0173)	ND(0.0414)	ND(0.0173)	ND(0.0173)	ND(0.0207)	ND(0.0173)	ND(0.0173)	ND(0.0345)	ND(0.0156)	ND(0.0173)	ND(0.0173)	ND(0.0173)	ND(0.0173)
8/30/84															
Nozzle BA	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0035)	ND(0.0098)	ND(0.0098)	ND(0.0098)	ND(0.0098)
Nozzle BA Field Blank	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0107)	ND(0.0044)	ND(0.0044)	ND(0.0044)	ND(0.0044)
Nozzle BB #1	21.8	10.3	ND(0.537)	0.437	ND(0.537)	ND(0.537)	ND(0.537)	ND(0.537)	ND(0.537)	0.437	ND(0.0416)	ND(0.0416)	ND(1.07)	ND(0.537)	ND(0.537)
Nozzle BB #1 Field Duplicate	19.3	11.4	ND(0.946)	ND(0.946)	ND(0.946)	ND(0.946)	ND(0.946)	ND(0.946)	ND(0.946)	ND(1.42)	ND(0.0603)	ND(1.42)	ND(1.42)	ND(0.946)	*
Nozzle BB #2	ND(0.0450)	ND(0.0277)	ND(0.0277)	ND(0.0277)	ND(0.0277)	ND(0.0277)	ND(0.0277)	ND(0.0277)	ND(0.0277)	ND(0.0277)	ND(0.0349)	ND(0.0208)	ND(0.0277)	ND(0.0277)	*
Nozzle BB #2 Field Duplicate	0.510	0.186	ND(0.0258)	0.0206	ND(0.0258)	ND(0.0129)	ND(0.0129)	ND(0.0129)	ND(0.0129)	ND(0.0361)	ND(0.0122)	ND(0.0258)	ND(0.0361)	ND(0.0258)	ND(0.0258)
Nozzle C	39.9	20.4	ND(1.00)	ND(2.01)	ND(2.01)	ND(2.01)	ND(2.01)	ND(1.00)	ND(1.00)	ND(2.01)	ND(0.152)	ND(2.01)	ND(2.01)	ND(2.01)	ND(2.01)
Nozzle C Field Duplicate	8.84	10.8	ND(0.270)	0.431	ND(0.270)	ND(0.270)	ND(0.270)	ND(0.270)	ND(0.270)	1.40	ND(0.180)	0.323	*	*	*
Nozzle C Field Blank	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)	ND(0.0394)
9/5/84															
Nozzle BA	ND(0.0015)	ND(0.0013)	ND(0.0013)	ND(0.0013)	ND(0.0013)	ND(0.0013)	ND(0.0013)	ND(0.0013)	ND(0.0013)	ND(0.0013)	ND(0.0094)	ND(0.0013)	ND(0.0013)	ND(0.0013)	ND(0.0013)
Nozzle BA Field Blank															
Nozzle BB	4.05	1.84	ND(0.200)	ND(0.200)	ND(0.200)	ND(0.200)	ND(0.200)	ND(0.200)	ND(0.200)	ND(0.200)	ND(0.0224)	ND(0.200)	ND(0.200)	ND(0.200)	ND(0.200)
Nozzle BB Lab Duplicate	4.16	1.55	ND(0.101)	0.084	ND(0.101)	ND(0.101)	ND(0.101)	ND(0.101)	ND(0.101)	0.084	ND(0.0214)	ND(0.202)	ND(0.202)	ND(0.101)	ND(0.101)
Nozzle C	0.636	0.199	ND(0.0660)	ND(0.0660)	ND(0.0660)	ND(0.0660)	ND(0.0660)	ND(0.0660)	ND(0.0660)	ND(0.0660)	ND(0.0796)	ND(0.0660)	ND(0.0660)	ND(0.0660)	ND(0.0660)
Nozzle C Field Blank															

NOTE: Data expressed in ng/g.

a. Volatile Compounds

Field blank samples were found to contain relatively low levels of compounds not generally found in samples of wastewater. As with semi-volatile compounds, no volatiles were detected in first-day samples at levels higher than those in the field blank. On the third sampling day, however, several target compounds were detected, though not all were found in both actual and field duplicate samples. A summary of these data appears below:

Table D-21

Low-BTU Liquid Waste
Target Volatile Compounds Detected
9/5/84

Compound	Concentration (ug/L) Sample	Concentration (ug/L) Field Duplicate	Precision (RPD)
1,1-dichloroethylene (vinylidene chloride)	127	137	3.8
1,1-dichloroethane (ethylene dichloride)	86	93	7.8
Chloroform	12	13	8.0
Tetrachloroethylene (perchloroethylene)	378	ND*	--
Monochlorobenzene	260	ND	--
Carbon tetrachloride	ND	2916	--
Trichloroethylene	ND	8	--

* ND = Not detected.

As shown in detail in Table D-23, other compounds were found in dike water samples from the third sampling day, summarized as follows:

Table D-22

Low-BTU Liquid Waste
Other Volatile Compounds Detected
9/5/84

Compound	Concentration (ug/L) Sample	Concentration (ug/L) Field Duplicate	Precision (RPD)
Methylene chloride	1127	1241	9.6
Acetone	1163	1302	11.3
1-(methylethyl)-benzene	2791	6222	76.1

The first two compounds commonly appear as contaminants in laboratory analyses; therefore, their presence in dike water may be questionable.

Accuracy criteria were met for all five samples (see Table D-23).

TABLE D-23
LOW-BTU LIQUID WASTE - VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

																TENTATIVELY IDENTIFIED COMPOUNDS			ACCURACY (% SURROGATE RECOVERY)				
	1,1,1-trichloroethane	benzene	toluene	ethylbenzene	styrene	methylene chloride	acetone	vinylidene chloride	ethylene dichloride	chloroform	perchloroethylene	chlorobenzene	total xylenes	carbon tetrachloride	trichloroethylene	hexamethylcyclo-trisiloxane	1-(methylethyl)-benzene	propylbenzene	toluene - D8	bromofluorobenzene	1,2 - dichloroethane - D4		ACCEPTABLE[1]
8/28/84																							
COMPOSITE SAMPLE						6													102	84	100		YES
FIELD BLANK						170													105	114	95		YES
9/5/84																							
COMPOSITE SAMPLE						1127	1163	127	86	12	378	260	429				2791	1160	101	95	77		YES
FIELD DUPLICATE		91				1241	1302	137	93	13			7659	2916	8		6222		101	104	119		YES
FIELD BLANK	24	3	4	14	12											200			98	102	106		YES

NOTE: Data expressed in ug/L.
[1] All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.

COMPLETENESS = 5/5 = 100%

b. Semi-Volatile Compounds

Field blank samples taken on both days on which dike water was fed to the incinerator were generally free of detectable contamination; a phthalate was noted in one of the blanks. No detectable semi-volatile compounds were found in the composite water sample taken on the first day. On the third day, 1,2-dichlorobenzene, 2-methylnaphthalene, 1,4-dichlorobenzene, and 2,4,6-trichlorophenol were detected, but only in the field duplicate sample in the case of the latter two. Following is a summary of these results:

Table D-24

Low-BTU Liquid Waste
Semi-Volatile Compounds Detected
9/5/84

	Concentration (ug/L)		
Compound	Sample	Field Duplicate	Precision (RPD)
1,2-dichlorobenzene	121	95	24.1
2-methylnaphthalene	174	809	129.2
1,4-dichlorobenzene	ND*	313	--
2,4,6-trichlorophenol	ND	167	--

* ND = Not detected.

The presence of 1,2-dichlorobenzene in this wastewater stream on the third day is affirmed within satisfactory bounds of precision. At the flow rate described above, this concentration corresponds to a mass flow of 0.34 to 0.44 milligram per day of 1,2-dichlorobenzene to the incinerator on the third sampling day. While 2-methylnaphthalene was present, the analytical data failed to meet established limits for precision.

Other tentatively identified compounds (Table D-25), some of them possibly of interest as they are ring compounds, were detected in the sample but not the field duplicate, and vice versa, on the third day. In the single case in which a compound was found in both (a substituted naphthalene), quality assurance criteria for precision were not met. Accuracy goals, as measured by recovery of base-neutral and acid surrogate compounds, were met for four of the five samples in this category, resulting in completeness of 80% (see Table D-25). Recovery of an acid surrogate was outside of the acceptable range in the sample not meeting the accuracy criterion.

c. PCDD/PCDF

(1) All Homologues

These data are presented in Tables D-26 and D-27. When compared to the liquid waste feeds data in Section B.1.d., the concentrations of PCDD/PCDF

TABLE D-25
LOW-BTU LIQUID WASTE - SEMI-VOLATILE COMPOUNDS[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

	QUANTITATED COMPOUNDS					TENTATIVELY - IDENTIFIED COMPOUNDS										ACCURACY (% SURROGATE RECOVERY)							
																BASE-NEUTRALS			ACIDS				
	1,2-dichlorobenzene	2-methylnaphthalene	bis(2-ethylhexyl)phthalate	1,4-dichlorobenzene	2,4,6-trichlorophenol	2,4-dimethyl-2,3-heptadiene -5-yne	propylbenzene	undecane	1,4-dihydro-1,4-methano-naphthalene	2-12-(2-butoxyethoxy) ethoxyl-ethanol	1-phenyl-1,2-ethanediol	1,4-dimethylbenzene	1-ethenyl-3-methylbenzene	1,3-benzenedicarboxylic acid, methylester	1,1-biphenyl-2-ol	nitrobenzene - D5	2-fluorobiphenyl	terphenyl - D14	phenol - D5	2-fluorophenol	2,4,6-tribromophenol		ACCEPTABLE[2]
8/28/84																							
Composite Sample																79	80	124	58	76	97		YES
Field Blank																58	70	86	16	50	97		NO
9/5/84																							
Composite Sample	121	174	49			13	6	29	62	52	1051					56	48	53	56	63	76		YES
Field Duplicate	95	809	96	313	167				1807			461	591	2398	4368	79	45	50	29	75	27		YES
Field Blank																49	48	53	31	54	55		YES

	Base-Neutrals	Acids	Overall
COMPLETENESS[3]	100%	80%	80% (4/5)

Notes: [1] Data expressed in ug/L.

[2] All surrogate recoveries within target range (20-180%) established in Quality Assurance Project Plan.

[3] By class of surrogates (acids and base-neutrals) and overall (combined).

TABLE D-26
LOW-BTU LIQUID WASTE - PCDD/PCDF ANALYSES[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

													Accuracy (% Surrogate Recovery)			
SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	$^{13}C_{12}$2378-TCDD	$^{37}Cl_4$2378-TCDD	$^{13}C_{12}$OCDD	$^{37}Cl_4$2378-TCDF
8/28/84																
COMPOSITE SAMPLE				10.4									60	114	100	39
FIELD BLANK													62	93	100	43
8/30/84																
(NO SAMPLE TAKEN - Low-BTU liquid waste was not incinerated on this day)																
9/5/84																
COMPOSITE SAMPLE		29.3			181	753		33.9					45	93	100	37
FIELD DUPLICATE		22.8			132	570		46.4					84	78	100	63
FIELD BLANK													100	108	96	46
PRECISION(RPD) - SAMPLE AND FIELD DUPLICATE		25			31	28		31								
									COMPLETENESS BY SURROGATE				80%	100%	100%	20%

NOTES:
1. All data expressed in pg/g.
2. All surrogate recoveries within target range of 50-150%.
3. Blank spaces denotes homologue not detected. Detection limits ranged from 0.3-3 ppq for TCDD and TCDF, to 14-28 ppq for OCDD and OCDF.

TABLE D-27
LOW-BTU LIQUID WASTE - PCDD/PCDF ANALYSES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
8/28/84 COMPOSITE SAMPLE	ND (.0008)	ND (.0010)	ND (.0024)	ND (.0031)	0.0104	ND (0.162)	ND (.0003)	ND (.0010)	ND (.0011)	ND (.0016)	ND (.0061)	ND (.0098)
FIELD BLANK	ND (.0009)	ND (.0010)	ND (.0026)	ND (.0027)	ND (.0068)	ND (.0089)	ND (.0005)	ND (.0010)	ND (.0013)	ND (.0051)	ND (.0036)	ND (.0104)
9/5/85 COMPOSITE SAMPLE	ND (.0102)	0.0293	ND (.0024)	ND (.0028)	0.181	0.753	ND (.0007)	0.0339	ND (.0016)	ND (.0015)	ND (.0040)	ND (.0047)
FIELD DUPLICATE	ND (.0027)	0.0228	ND (.0035)	ND (.0055)	0.132	0.570	ND (.0009)	0.0464	ND (.0036)	ND (.0081)	ND (.0143)	ND (.0280)
FIELD BLANK	ND (.0002)	ND (.0010)	ND (.0010)	ND (.0029)	ND (.0067)	ND (.0138)	ND (.0003)	ND (.0010)	ND (.0046)	ND (.0025)	ND (.0013)	ND (.0166)

Notes: Data expressed in ng/g. Accuracy data appear in Table D-26.

are low, as may be expected, and limited to the tetra-, hepta-, and octa-CDD, and tetra-CDF homologues. On the third sampling day, OCDD comprised approximately 78% by weight of the PCDD detected.

Note, however, that none of the five sample analyses met the accuracy criteria established for the four surrogate compounds. These data are therefore not suitable for quantitative purposes, despite the good precision observed (see Table D-26).

(2) TCDD Isomers

No 2378-TCDD or 2378-TCDF were detected. The isomers found (see Tables D-28 and D-29) were limited to 1368, 1378, and 1237/1238.

D. Incinerator Exhaust

1. Volatile Compounds

Method blanks of the Tenax and charcoal adsorbents appeared to contain measurable amounts of several contaminants; however, the analytical accuracy may be questionable as none of the four surrogates was recovered. Analytical problems traceable to poor fit of the VOST tubes in the thermal desorbing unit were frequently cited by the laboratory. This phenomenon usually first affected the recovery of the surrogate 1,2-dichloroethane-D_4, but not of the other three surrogates. The data summaries (Tables D-30, D-31, and D-32) indicate the data points which were affected in this way.

On the three sampling days, five or six sets of VOSTs were exposed, usually for 40 minutes, with single field blanks for the sorbents and condensates on each day covering the time period in which all five or six sets were operated. Thus, where compounds were detected in the field blank, the amounts trapped were apportioned to each exposed sample according to the length of sampling time. For example, if six VOSTs were employed for 40 minutes each, resulting in a total sampling time of 240 minutes, one-sixth of the amount of a compound in the field blank was subtracted from that in each exposed sample.

In Tables D-30, D-31, D-32, no data are stated for compounds trapped in the collected condensates, which were pooled in the field and analyzed as composites of all of the sample runs on each day. With few exceptions (see raw data, Tables D-33, D-34, D-35), no compounds other than methylene chloride and acetone were detected, and it is suspected that these findings may be the result of typical laboratory contamination.

The data tables present the compounds detected in terms of those specifically targeted, other chlorinated compounds, benzene ring compounds, and other ring compounds. While the materials consumed in the 703 Building incinerator varied from day to day, it is apparent that carbon tetrachloride and 1,4-dichlorobenzene were present in exhaust gas almost continuously. Other compounds, such as 1,2-dichlorobenzenes, ethylbenzene, 1,1,1-trichloroethane, and toluene, were found in measurable concentrations but not on all three sampling days.

TABLE D-28
LOW-BTU LIQUID WASTE - TCDD ISOMERS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
8/28/84															
COMPOSITE SAMPLE															
FIELD BLANK															
8/30/84															
(NO SAMPLE TAKEN -	Low-BTU liquid waste was not incinerated on this day.)														
9/5/84															
COMPOSITE SAMPLE	16.2	5.3								3.4					
FIELD DUPLICATE	12.0	6.0								2.0					
FIELD BLANK															
PRECISION(RPD) - SAMPLE AND FIELD DUPLICATE	29.8	12.4								51.9					

Notes: Data expressed in pg/g. Blank spaces denote isomer was not detected.

TABLE D-29
LOW-BTU LIQUID WASTE - TCDD ISOMERS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
8/28/84 COMPOSITE SAMPLE	ND (.0017)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0008)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
FIELD BLANK	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0009)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
9/5/84 COMPOSITE	0.0162	0.0053	ND (.0011)	ND (.0011)	ND (.0010)	ND (.0011)	ND (.0011)	ND (.0010)	ND (.0010)	0.0034	ND (.0102)	ND (.0011)	ND (.0011)	ND (.0010)	ND (.0011)
FIELD DUPLICATE	0.0120	0.0050	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	0.0020	ND (.0027)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
FIELD BLANK	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0002)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)

NOTE: Data expressed in ng/g.

TABLE D-30
INCINERATOR EXHAUST VOLATILE COMPOUNDS AS MEASURED USING VOLATILE ORGANIC SAMPLING TRAIN, IN TERMS OF CONCENTRATION IN AIR
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

		TARGET COMPOUNDS									OTHER CHLORINATED COMPOUNDS					BENZENE RING COMPOUNDS				OTHER RING COMPOUNDS				ACCURACY (% SURROGATE RECOVERY)				
SAMPLE ID	UNITS	CARBON TETRACHLORIDE	MONOCHLOROBENZENE	DICHLOROBENZENE*	1,2-DICHLOROBENZENE*	1,3-DICHLOROBENZENE*	1,4-DICHLOROBENZENE*	CHLOROFORM**	PERCHLOROETHYLENE	TRICHLOROETHYLENE	1,1,1-TRICHLOROETHYLENE	1,1,2-TRICHLOROETHANE	1,2-DICHLOROPROPANE	1,2,3-TRICHLOROPROPANE*	DIBROMOCHLOROMETHANE	BENZENE	ETHYLBENZENE	STYRENE	TOLUENE	METHYLCYCLOPENTANE*	1,3-CYCLOPENTADIENE*	1,3,5-CYCLOHEPTATRIENE*	METHYLCYCLOHEXANE*	TOLUENE - D8	BROMOFLUOROBENZENE	1,2-DICHLOROETHANE - D4	ETHYLBENZENE - D10	ACCEPTABLE[1]
FIRST FRONT TUBE	ug/m^3	161	92		190		204	6	10	--	33	10	54			42	51	--	104					118	114	58	92	NO
FIRST BACK TUBE	ug/m^3	23	--		--		55	17	--	--	6	--				2	3	--	17					80	68	70	122	NO
FIELD DUPLICATE FRONT TUBE	ug/m^3		(Sample analysis not returned from laboratory.)																									
FIELD DUPLICATE BACK TUBE	ug/m^3		(Sample analysis not returned from laboratory.)																									
SECOND FRONT TUBE	ug/m^3	133	151		--		351	17	17	7	--	8	40			--	53	--	125					96	82	70	102	NO
SECOND BACK TUBE	ug/m^3	144	--		277		377	25	3	4	--	--				13	62	--	94					104	88	98	90	YES
THIRD FRONT TUBE	ug/m^3	188	104		--		295	12	16	9	20	--	47			18	36	171	138					100	80	118	82	YES
THIRD BACK TUBE	ug/m^3	17	--		--		--	--	--	--		--				2	--	--	--					114	98	0	96	NO
FOURTH FRONT TUBE	ug/m^3	243	133		522		471	ND	15	10	335	--	48	8		36	102	--	174					86	88	6	86	NO
FOURTH BACK TUBE	ug/m^3	--	--		--		89	--	--	--	190	--				7	1	--	--					78	138	0	100	NO
FIFTH FRONT TUBE	ug/m^3	--	--		ND		--	ND			--	--				--	--	2	ND					67	108	133	75	NO
FIFTH BACK TUBE	ug/m^3	--	--		--		--	--			--	--				--	--		--					72	128	10	153	NO
SIXTH FRONT TUBE	ug/m^3		(Sample analysis not returned from laboratory.)																									
SIXTH BACK TUBE	ug/m^3																											
FIELD BLANK																												
FRONT TUBE	ng	1676			4231		4627	820	62	80	759					108	802		1089					94	84	72	112	NO
BACK TUBE	ng	135						20			102					32	200		236					92	84	600	100	NO

COMPLETENESS = 2/16 = 13%

Notes - [1] All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.
* Tentatively-identified compound.
** Breakthrough volume exceeded during sampling.
ND Compounds present on blank tubes in higher concentrations than on exposed sample.

TABLE D-31
INCINERATOR EXHAUST VOLATILE COMPOUNDS AS MEASURED USING VOLATILE ORGANIC SAMPLING TRAIN, IN TERMS OF CONCENTRATION IN AIR
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

SAMPLE ID	UNITS	TARGET COMPOUNDS									OTHER CHLORINATED COMPOUNDS					BENZENE RING COMPOUNDS				OTHER RING COMPOUNDS				ACCURACY (% SURROGATE RECOVERY)				
		CARBON TETRACHLORIDE	MONOCHLOROBENZENE	DICHLOROBENZENE*	1,2-DICHLOROBENZENE*	1,3-DICHLOROBENZENE*	1,4-DICHLOROBENZENE*	CHLOROFORM**	PERCHLOROETHYLENE	TRICHLOROETHYLENE	1,1,1-TRICHLOROETHYLENE	1,1,2-TRICHLOROETHANE	1,2-DICHLOROPROPANE	1,2,3-TRICHLOROPROPANE*	DIBROMOCHLOROMETHANE	BENZENE	ETHYLBENZENE	STYRENE	TOLUENE	METHYLCYCLOPENTANE*	1,3-CYCLOPENTADIENE*	1,3,5-CYCLOHEPTATRIENE*	METHYLCYCLOHEXANE*	TOLUENE - D8	BROMOFLUOROBENZENE	1,2 DICHLOROETHANE - D4	ETHYLBENZENE - D10	ACCEPTABLE[1]
FIRST FRONT TUBE	ug/m^3	--	15	--	--	--	1234	--	5	--	--	--	--		--	--	13	--	--	9		--	--	100	112	2	78	NO
FIRST BACK TUBE	ug/m^3	34	--	10	38	56	59	--	--	1		--	--		--	--	6	--	41	--		--	113	82	120	5	80	NO
FIELD DUPLICATE FRONT TUBE	ug/m^3	258	--	6	--	--	--	--	--	11		11	58		34	--	--	--	--	--		--	--	104	82	4	92	NO
FIELD DUPLICATE BACK TUBE	ug/m^3	--	--	--	--	--	ND	--	--	--		--	--		--	--	--	--	--	--		--	--	64	100	28	24	NO
SECOND FRONT TUBE	ug/m^3	--	--	--	--	--	284	--	--	--		--	--		--	--	1	7	ND	--		--		94	96	16	0	NO
SECOND BACK TUBE	ug/m^3	--	--	--	--	--	--	--	--	--		--	--		--	--	--	--	ND	--		9		122	88	18	80	NO
THIRD FRONT TUBE	ug/m^3	201	95	--	--	--	284	101	28	5			23		--	--	--	--	--	--		--		116	70	2	100	NO
THIRD BACK TUBE	ug/m^3	85	--	--	725	--	--	5	--	6			--		--	--	57	--	256	--				92	96	30	86	NO
FOURTH FRONT TUBE	ug/m^3	532	64	--	422	--	250	9	7	14			71			92	67	--	115	2041				80	92	26	48	NO
FOURTH BACK TUBE	ug/m^3	--	--	--	--	--	--	--	--	--			--		--	ND	--	--	--	--				0	0	0	0	NO
FIFTH FRONT TUBE	ug/m^3	323	465	--	3812	--	2673	5	68	6			48			47	--	--	545	3782				98	112	14	80	NO
FIFTH BACK TUBE	ug/m^3	262	--	--	--	--	303	--	--	9			--			--	30	--	225	--				0	0	18	0	NO
SIXTH FRONT TUBE	ug/m^3	375	5	--	--	--	--	--	--	6			14			32	4	--	--	--				0	0	0	0	NO
SIXTH BACK TUBE	ug/m^3	--	--	--	--	--	7	--	--	--			--			--	ND	--	ND	--				0	0	0	0	NO
FIELD BLANK																												
FRONT TUBE	ng							5											548					58	58	14	174	NO
BACK TUBE	ng						2058									1238	210		1385	2476				70	112	0	100	NO

COMPLETENESS = 0/16 = 0%

Notes - [1] All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.
* Tentatively-identified compound.
** Breakthrough volume exceeded during sampling.
ND Compounds present on blank tubes in higher concentrations than on exposed sample.

TABLE D-32
INCINERATOR EXHAUST VOLATILE COMPOUNDS AS MEASURED USING VOLATILE ORGANIC SAMPLING TRAIN, IN TERMS OF CONCENTRATION IN AIR
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

		TARGET COMPOUNDS									OTHER CHLORINATED COMPOUNDS					BENZENE RING COMPOUNDS				OTHER RING COMPOUNDS				ACCURACY (% SURROGATE RECOVERY)				
SAMPLE ID	UNITS	CARBON TETRACHLORIDE	MONOCHLOROBENZENE	DICHLOROBENZENE*	1,2-DICHLOROBENZENE*	1,3-DICHLOROBENZENE*	1,4-DICHLOROBENZENE*	CHLOROFORM**	PERCHLOROETHYLENE	TRICHLOROETHYLENE	1,1,1-TRICHLOROETHYLENE	1,1,2-TRICHLOROETHANE	1,2-DICHLOROPROPANE	1,2,3-TRICHLOROPROPANE*	DIBROMOCHLOROMETHANE	BENZENE	ETHYLBENZENE	STYRENE	TOLUENE	METHYLCYCLOPENTANE*	1,3-CYCLOPENTADIENE*	1,3,5-CYCLOHEPTATRIENE*	METHYLCYCLOHEXANE*	TOLUENE - D8	BROMOFLUOROBENZENE	1,2-DICHLOROETHANE - D4	ETHYLBENZENE - D10	ACCEPTABLE[1]
FIRST FRONT TUBE	ug/m^3	120	94	--	98	--	57	64	36	10	--		21			--	26	123	--		6			73	55	101	40	NO
FIRST BACK TUBE	ug/m^3	--	--	--	--	--	14	--	--	--	--		--			--	--	--	--		--			94	94	72	78	NO
FIELD DUPLICATE FRONT TUBE	ug/m^3																											
FIELD DUPLICATE BACK TUBE	ug/m^3																											
SECOND FRONT TUBE	ug/m^3	142	--	--	--	--	225	--	38	8	--		47			--	46	--	--					74	66	74	116	NO
SECOND BACK TUBE	ug/m^3	28	--	22	--	--	1089	--	--	--	--		--			--	--	--	--					78	82	127	124	NO
THIRD FRONT TUBE	ug/m^3	--	--	--	--	--	202	--	--	--	--		--			--	--	--	--					90	100	136	124	NO
THIRD BACK TUBE	ug/m^3	226	--	--	196	34	282	5	--	--	--		--			40	44	--	--					86	92	28	82	NO
FOURTH FRONT TUBE	ug/m^3	--	--	--	--	--	--	--	--	--	--		68			--	38	--	--					106	76	16	146	NO
FOURTH BACK TUBE	ug/m^3	--	--	--	--	--	78	--	--	--	--		--			--	--	--	--					100	94	86	146	NO
FIFTH FRONT TUBE	ug/m^3	128	--	--	--	--	--	9	15	4	--		28			--	--	--	--					110	108	32	88	NO
FIFTH BACK TUBE	ug/m^3	--	--	--	--	--	192	--	--	--	--		--			18	--	--	--					94	90	96	10	NO
SIXTH FRONT TUBE	ug/m^3	97	58	--	333	--	21	4	14	4	--		11			--	45	--	--					94	84	36	106	NO
SIXTH BACK TUBE	ug/m^3	--	--	--	--	--	--	--	--	--	--		--			--	--	--	ND					82	114	42	92	NO
FIELD BLANK																												
FRONT TUBE	ng	1160					2513			79						419	747		1840					86	92	0	64	NO
BACK TUBE	ng	351			556	669										237	216		306					76	104	82	84	NO

COMPLETENESS = 0/16 = 0%

Notes - [1] All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.
* Tentatively-identified compound.
** Breakthrough volume exceeded during sampling.
ND Compounds present on blank tubes in higher concentrations than on exposed sample.

TABLE D-33
INCINERATOR EXHAUST VOLATILE COMPOUNDS AS MEASURED USING VOLATILE ORGANIC SAMPLING TRAIN
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

		QUANTITATED COMPOUNDS																TENTATIVELY IDENTIFIED COMPOUNDS											
	UNITS	METHYLENE CHLORIDE	TOLUENE	ETHYLBENZENE	STYRENE	TOTAL XYLENES	CHLOROFORM*	2-BUTANONE	1,1,1-TRICHLOROETHANE	CARBON TETRACHLORIDE*	1,2-DICHLOROPROPANE	1,1,2-TRICHLOROETHANE	BENZENE	PERCHLOROETHYLENE*	CHLOROBENZENE*	TRICHLOROETHYLENE*	ACETONE	DICHLOROBENZENE*	1,1,2 - TRICHLORO - 1,2,2 - TRIFLUOROETHANE	1,4 - DICHLOROBENZENE*	1,2 - DICHLOROBENZENE*	HEXANE	HEXAMETHYLCYCLOTRISILOXANE	DIMETHOXYMETHANE	1,2,3 - TRICHLOROPROPANE	HEXAMETHYLDISILOXANE	PENTANE	DIMETHOXYDIMETHYLSILANE	1,3 - DICHLOROBENZENE*
METHOD BLANKS																													
VOST TENAX/TENAX-Charcoal	ng	18000	428	344	264	295														831		2200	200					800	
VOST CONDENSER	ug/L	3518																											
VOST TUBES																													
FIRST FRONT TUBE	ng		2152	1191		1016	282	1115	818	3571	1087	201	873	198	1870				670	5040	4664								
FIRST BACK TUBE	ng		857	105		482			132	572			46							1106									
SECOND FRONT TUBE	ng	11	2524	1256		714	514			2966	816	147		368	3054	160				8038		5746	200						
SECOND BACK TUBE	ng		1791	1337		11925	510			2989			282	52		16				7646	5630								
THIRD FRONT TUBE	ng		2766	909	3144	731	280		560	4186	950		392	338	2149	193				9671		850	200						
THIRD BACK TUBE	ng	55								462			44				346						1200			200			
FOURTH FRONT TUBE	ng		3478	2334		1360	97		7738	5226	1027		172	332	2667	237				10388	11344	860		175	150				
FOURTH BACK TUBE	ng	157		270		1511			4039				154							1804									
FIFTH FRONT TUBE	ng		28		44		66														36								
FIFTH BACK TUBE	ng	1427																									4000		
FIELD BLANK (Composite of 5 Runs)	ng		1089 236	802 200		9482 811	820 20	440	759 102	1676 135			108 32	62		80				4627	4231	1784	200 3244						
VOST CONDENSER																													
COMPOSITE SAMPLE (5 Runs)	ug/L	122															9198												
FIELD BLANK	ug/L	49															5571												

* Target Volatile Compound

TABLE D-34
INCINERATOR EXHAUST VOLATILE COMPOUNDS AS MEASURED
USING VOLATILE ORGANIC SAMPLING TRAIN
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

		QUANTITATED COMPOUNDS																	TENTATIVELY IDENTIFIED COMPOUNDS													
	UNITS	METHYLENE CHLORIDE	TOLUENE	ETHYLBENZENE	STYRENE	TOTAL XYLENES	CHLOROFORM*	2-BUTANONE	CARBON TETRACHLORIDE*	1, 2-DICHLOROPROPANE	1,1,2-TRICHLOROETHANE	BENZENE	TETRACHLOROETHYLENE*	CHLOROBENZENE*	TRICHLOROETHYLENE*	ACETONE	DIBROMOCHLOROMETHANE	BROMOFORM	1,4-DICHLOROBENZENE*	1,2-DICHLOROBENZENE*	HEXANE	HEXAMETHYLCYCLOTRISILOXANE	DIBROMOMETHANE	METHYLCYCLOPENTANE	TRICHLOROFLUOROMETHANE	3-METHYLPENTANE	BENZOFURAN	DICHLOROBENZENE*	1,3,5-CYCLOHEPTATRIENE	1,3-DICHLOROBENZENE*	METHYLCYCLOHEXANE
VOST TUBES																															
FIRST FRONT TUBE	ng			251		70							88	284					24,900				92	183							
FIELD DUPLICATE FRONT TUBE	ng	215					1168		4997	1131	215				213		648	1992					284					187			
FIRST BACK TUBE	ng		1028	149		595		840	655						23				1371	748		200				1990			1094		2190
FIELD DUPLICATE BACK TUBE	ng					112													136												
SECOND FRONT TUBE	ng		83	27	136																19,413				1183		50	133			
SECOND BACK TUBE	ng		190																			200							178		
THIRD FRONT TUBE	ng					1163	1884		3754	438			526	1773	95				5309												
THIRD BACK TUBE	ng		5015	1112		1640	93	4479	1593						104					13559											
FOURTH FRONT TUBE	ng		2245	1253		544	170		4972	1325		1722	140	1198	272				4695	7920		200		38284							
FOURTH BACK TUBE	ng											61										200									
FIFTH FRONT TUBE	ng		10175			4678	96		5982	889		886	1262	8616	116				49484	70563				70000							
FIFTH BACK TUBE	ng		4389	600		230			4852						158				5841												
SIXTH FRONT TUBE	ng		522	67		23			7082	273		612		97	107																
SIXTH BACK TUBE	ng		78	35		68													365			200									
FIELD BLANK			548 1385	210		6328 362	54					1238							2058			200		2476		1857					
VOST CONDENSER																															
COMPOSITE SAMPLE	ug/L	83														439					10										
FIELD DUPLICATE	ug/L	109														1810															
FIELD BLANK	ug/L	871														943					20										

*Target volatile compound.

TABLE D-35
INCINERATOR EXHAUST VOLATILE COMPOUNDS AS MEASURED
USING VOLATILE ORGANIC SAMPLING TRAIN
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

		QUANTITATED COMPOUNDS													TENTATIVELY IDENTIFIED COMPOUNDS														
	UNITS	Methylene chloride	Toluene	Ethylbenzene	Styrene	Total xylenes	Chloroform*	Carbon tetrachloride*	1,2 - dichloropropane	Benzene	Perchloroethylene*	Chlorobenzene*	Trichloroethylene*	Acetone	1,4 - dichlorobenzene*	1,2 - dichlorobenzene*	Hexane	Hexamethylcyclotrisiloxane	Oxisane	2 - (methoxyethyl) - trimethylsilane	Trichlorofluoromethane	3 - methylpentane	Hexamethyldisiloxane	Dichlorobenzene*	Fluorotrimethylsilane	2,4 - dimethyl-1-pentene	1,3 - dichlorobenzene*	2 - methylpentene	1,3 - cyclopentadiene
VOST Tubes																													
First Front Tube	ng			638	2453	463	1272	2586	412		717	1863	205		1557	1944													117
First Back Tube	ng									36					278			200				100	500						
Second Front Tube	ng			1000		574		2915	896		762		165		4728														
Second Back Tube	ng	61						545										200						429	1500	500			
Third Front Tube	ng														20560														
Third Back Tube	ng			1036		4855	100	4532		967					3735	4173						9711					3137		
Fourth Front Tube	ng			866					905						4165														
Fourth Back Tube	ng					50		314		44								200								785			
Fifth Front Tube	ng						174	2762	539		295		98		1944														
Fifth Back Tube	ng									561								200				50							
Sixth Front Tube	ng		192	1016		1083	77	2104	220		280	1136	98	1354	4195	6550													
Sixth Back Tube	ng			92		526				91					410			200			9658	873							
Field Blank			1840	747		1033		1160		419			79		2513			200				1150							
(Composite of 6 Runs)	ng		306	216		300		351		237						556	810	200				1100					669	1850	
VOST Condenser																													
Composite Sample (6 Runs)	ug/L	28												398					16	14									
Field Blank	ug/L	140												950															

* Target Volatile Compound

The distribution of these compounds appeared generally random between the front and back detection tubes when measurable quantities were found in each. Also, several cases appear in which the same compound was detected only in the front tube during one run, and only in the back tube in another. Field duplicate samples taken during the first of six runs on the second sampling day were not comparable for any compound, and no evaluation of precision could be made. Field blank samples showed no clear, consistent pattern of blank contamination; in some cases, denoted by the label "ND", compounds were present in higher concentrations on blanks than on exposed samples.

For screening purposes, these data, despite their variability, appear to suggest strongly that several compounds were regularly present in exhausts from the 703 Building incinerator stack. However, any contributions of organics resulting from the venting of gases from the activated carbon bed filter serving the liquid waste tank storage area cannot be assessed from these data. Therefore, emissions of the above compounds cannot be differentiated according to their source.

A detection limit goal of 1 ppb was set with respect to collection of volatile compounds using the VOST. Actual method sensitivities were 0.25 to 0.50 microgram/cubic meter, and indicated the objective was met. However, the completeness goal of 90% was not met, as only 5% of the samples submitted were analyzed such that surrogate recoveries were within the acceptable range of 80 to 125% (see Tables D-30, D-31, and D-32).

2. Semi-Volatile Compounds

Method blanks of the glass fiber filter, XAD-2 sorbent, and impinger catch (distilled, deionized water) were found free of contaminants other than two phthalates commonly considered ubiquitous in laboratory analyses.

The physical limitations of the sampling site at the incinerator outlet, and the need to sample simultaneously at the same location for PCDD/PCDF, precluded taking field duplicate samples to judge precision. In any event, sampling of incinerator exhaust gases for semi-volatiles revealed few compounds in detectable concentrations, with the exception of three base-neutral chlorobenzenes and naphthalene found only on the second day of sampling. As the recovery of the base-neutral surrogates in the XAD-2 sample was within the acceptable range (see Table D-36), these findings are supportable; however, it is suspected that breakthrough volumes for dichlorobenzenes on XAD-2 were exceeded in this sample. Therefore these data may be biased low. None of these compounds was found in any component of the Modified Method 5 train other than the primary XAD-2 sorbent cartridge. However, the presence of several substituted benzene, naphthalene, and phenyl compounds in the field blank sample on this day may point to contamination during sampling.

The chlorobenzene concentrations found on the second day of sampling, shown below, correspond to daily emissions of the following quantities of the listed compounds:

TABLE D-36

QUANTITATED AND TENTATIVELY IDENTIFIED SEMI-VOLATILE COMPOUNDS DETECTED IN INCINERATOR EXHAUST
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR - 8/28, 8/30, and 9/5/84

																															ACCURACY (% SURROGATE RECOVERY)						
																															Base-Neutrals			Acids			
	1,2-DICHLOROBENZENE	1,4-DICHLOROBENZENE	NAPHTHALENE	BENZOIC ACID	ISOPHORONE	BIS(2-ETHYLHEXYL) PHTHALATE	DI-N-OCTYL PHTHALATE	DI-N-BUTYL PHTHALATE	DIETHYL PHTHALATE		TETRACHLORO-BENZENE	BIPHENYL	1,4-DIMETHOXY-BENZENE	3,5-DIMETHOXY-2-CYCLOHEXENE-1-ONE	BENZOTHIAZOLE	2-ETHYL-1-HEXANOL	BUTYL-2-METHYL PROPYLPHTHALATE	1,3,5-TRITHIANE	1,4-DIHYDRO-1,4-ETHANONAPHTHALENE	2-ETHYL-1,1'-BIPHENYL	METHYL-DIPHENYLSILANE	1,1'-(1,2-ETHENDIYL)-BIS(Z)BENZENE	1,1':2',1"-TERPHENYL	TERPHENYL	1-NONANOL	3-METHYL-6-(1-METHYLENEI-DENE)-2-CYCLOHEXENE-1-ONE	7,7'-DICHLORO-BICYCLO-(4,1,0) HEPTANE	BIS-2-METHYL-PROPYLPHTHALATE	UNITS		NITROBENZENE-D5	2-FLUORO-BIPHENYL	TERPHENYL-D14	PHENOL-D5	2-FLUORO-PHENOL	2,4,6-TRIBROMO-PHENOL	ACCEPTABLE[1]
8/28/84																																					
Filter + Probe Wash						24																							ug/L		58	89	54	46	24	104	YES
Field Blank																													ug/L		47	80	35	65	49	60	YES
XAD-2 Cartridges																													ug/kg		0	0	0.2	0.3	0	0	NO
Field Blank						859		143																					ug/kg		110	273	133	91	20	0	NO
Impingers & Rinses						6.5																							ug/L		101	81	43	50	80	22	YES
Field Blank						13																							ug/L		70	91	29	101	25	20	YES
Backup XAD-2						67		422																					ug/kg		61	89	38	52	0	0	NO
Field Blank							4375	1537																					ug/kg		32	37	23	40	29	22	YES
8/30/84																																					
Filter + Probe Wash													4592	4353															ug/L		2240	120	204	2	0	0	NO
Field Blank					3120																								ug/L		36	59	21	27	22	53	YES
XAD-2 Cartridges	27540	24140	7820			9010		330			5900																		ug/kg		57	68	85	8	0	0	NO
Field Blank	219							1047											11027	5125	14680	7511	4403	5026					ug/kg		78	74	68	77	96	41	YES
Impingers & Rinses						30																							ug/L		56	85	47	48	41	56	YES
Field Blank																									1799	5098			ug/L		110	86	75	46	43	69	YES
Backup XAD-2				1600		428	476								633														ug/kg		55	64	52	35	79	46	YES
Field Blank						470		470																					ug/kg		61	80	77	40	85	48	YES
9/5/84																																					
Filter + Probe Wash												1875																	ug/L		7	34	56	0	0	0	NO
Field Blank																2300											1493		ug/L		34	71	71	0	0	0	NO
XAD-2 Cartridges																													ug/kg		0	0	1	0	0	0	NO
Field Blank						44																							ug/kg		50	30	84	0	0	0	NO
Impingers & Rinses						40																							ug/L		43	58	79	0	0	0	NO
Field Blank								2766																					ug/L		42	58	69	0	0	0	NO
Backup XAD-2						330		1466								27	18	3											ug/kg		64	97	42	56	85	25	YES
Field Blank																625												2020	ug/kg		91	111	44	51	43	20	YES
METHOD BLANKS																																					
Filter																															67	62	84	52	51	48	YES
Probe Wash																															10	5	0	0	0	0	NO
XAD-2 Cartridges						117			560																				ug/kg		71	77	122	71	57	86	YES
Impinger						69																							ug/L		91	68	106	64	71	100	YES

	Base-Neutrals	Acids	Overall
COMPLETENESS[2]	79%	57%	57% (16/28)

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[1] All surrogate recoveries within target range (20-180%) established in Quality Assurance Project Plan.

[2] By class of surrogates (acids and base-neutrals) and overall (combined).

Table D-37

Incinerator Exhaust
Semi-Volatile Compounds
8/30/84

Compound	Concentration (ug/m^3)	Daily Emissions (grams)
1,2-dichlorobenzene	115	141-150
1,4-dichlorobenzene	102	125-133
Tetrachlorobenzene	25	31-33

In addition, 40 to 43 grams per day of naphthalene (33 ug/m^3) were emitted from the incinerator at the operational level of the second sampling day.

Raw analytical data are presented in Table D-36; these show the presence of compounds not appearing on the target list (Table V-1). In the table, it is shown in completeness data that generally better accuracy (% surrogate recovery) was achieved for base-neutral compounds than for acid compounds. Phthalate compounds, considered to be common laboratory contaminants, were also frequently found. Also, substantial contamination by several compounds appeared in field blank samples, particularly on the second sampling day; however, though the surrogate recovery performance for those samples were generally acceptable for both acids and base-neutrals, those contaminants rarely appeared in the corresponding exposed samples. The detection limit objective of 5 ppb in air was achieved, with actual sensitivities on the order of 1 to 2 ug/m^3, or generally less than 1 ppb.

As indicated previously, recoveries of acid surrogates during analysis was frequently poor, especially from handling solid sorbent media or mixtures of solids and liquids. The recognized strong affinities to water exhibited by the phenolic (acid) surrogates may have been a factor in the poor observed recoveries, as the exhaust gases that passed through the sorbents were nearly saturated with moisture. In any event, overall completeness for this category of samples was 57% (16 of 28), including method blank samples (see Table D-36).

3. PCDD/PCDF

a. All Homologues

In Table D-40, these data show a full range of homologues were present in incinerator exhaust gases, particularly on the second sampling day. Tetra-homologues appeared to be present universally, while octa-homologues were also found frequently. Also of interest is the apparent tendency of most collected constituents to reside in the XAD-2 cartridge; however, the backup XAD-2 cartridges on the second and third sampling days were not analyzed successfully, and an evaluation of breakthrough was thus not possible on these days. Particularly high concentrations of TCDD and TCDF were found on the third day; however, accuracy measurements indicated these data to be questionable.

Overall completeness (see Table D-38), taking into account satisfactory accuracy and availability of data, did not meet the 90% goal established for this study. Detection limit criteria of 5 ppt for tetra-homologues and 15 ppt for other homologues, were met. The data in Table D-38 are restated in terms of concentration in air in Table D-39; also included is information with respect to the conditions placed on the summed data for the entire Modified Method 5 train, when precision, duplicate analysis, and spike analysis problems are considered. The analytical data presented in Table D-39 represent the total emissions of PCDD/PCDF homologues; these data were calculated by summing the homologues caught in each portion of the Modified Method 5 trains. Note from Table D-38 that some of these individual analyses were acceptable in terms of accuracy, while other analyses were marked by unacceptable surrogate recoveries.

b. TCDD Isomers

These data, presented in Table D-43 and in abridged form in Table D-41, are largely self-explanatory. When TCDD was present, the 1368 isomer was most common, followed by the 1379 and 1237/1238 isomers. No 2378-TCDD was detected in any of these samples. These data are restated in Table D-42 in terms of concentration in air.

4. Vinylidene Chloride

The results of instrumental (GC-ECD) analysis of Tedlar bag samples for vinylidene chloride are presented in Table D-44. Of the 20 samples obtained, all but one was analyzed in triplicate. Three individual data points were rejected as being much more than one standard deviation from the mean.

These data suggest that vinylidene chloride was present in exhaust gas continuously throughout the three sampling periods. However, analyses of the same samples by Dow Chemical using GC-MS indicated those peaks identified as vinylidene chloride were attributable to other compounds.

As with the VOST samples, the vent of the incinerator liquid waste tank farm activated carbon bed filter system was located upstream of the bag sampling location. Therefore, the compounds identified above may not be traceable entirely to emissions from the incinerator.

Table D-45 presents the results of a series of replicate analyses for vinylidene chloride carried out on the contents of the same Tedlar bags, approximately 24 hours apart. These data were intended to demonstrate possible changes in bag contents from the time of sampling until later analysis. The results appear to show random differences which are sufficiently small to indicate that delays between sampling and analysis did not cause significant errors.

TABLE D-38

INCINERATOR EXHAUST - PCDD/PCDF ANALYSES[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

													Accuracy (% Surrogate Recovery)			
SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	$^{13}C_{12}$2378-TCDD	$^{37}Cl_4$-2378-TCDD	$^{13}C_{12}$OCDD	$^{37}Cl_4$2378-TCDF
8/28/84																
Filter + Probe Wash		10.2				2.8	0.6	29.2	4.0	1.1			100	96	95	84
Field Blank													100	92	44	65
XAD-2 Cartridge		283	42.5	5.8	1.4	1.3	9.3	213	84.8	16.2	1.7	0.4	24	94	100	29
Field Blank								1.2					95	94	100	86
Impingers		16.6				2.3		287					100	93	49	94
Field Blank						2.2							100	93	61	90
Backup XAD-2		1.4						32.0					65	95	100	59
Field Blank													80	94	100	78
8/30/84																
Filter + Probe Wash		19.0	3.0	1.2	3.5	11.4	0.5	264	6.2	2.8	1.8	1.0	67	96	100	37
Field Blank													78	94	100	51
XAD-2 Cartridge		206	8.1			1.5	8.3	33.8	11.8	3.2			63	93	53	100
Field Blank													100	88	82	87
Impingers		24.4		0.9	1.3	1.5	0.7	141	6.3	5.1	1.4		58	96	100	51
Field Blank													100	97	54	84
Backup XAD-2		(Sample analysis not returned from laboratory.)														
Field Blank		(Sample analysis not returned from laboratory.)														
9/5/84																
Filter + Probe Wash		10.2				4.3		191	1.0				100	95	89	90
Field Blank		0.4				1.7		6.0					57	94	100	35
XAD-2 Cartridge		15.9					0.3	313					100	92	57	98
Field Blank													100	93	64	62
Impingers		2.1						36.1					100	90	53	87
Field Blank													72	108	100	44
Backup XAD-2		(Sample analysis not returned from laboratory.)														
Field Blank		(Sample analysis not returned from laboratory.)														
									COMPLETENESS BY SURROGATE				79%	83%	75%	67%

Notes:

1. Data expressed in nanograms per total sample. Detection limit data appear in Table D-40.
2. All surrogate recoveries within target range of 50-150%.

TABLE D-39

INCINERATOR EXHAUST - PCDD/PCDF ANALYSES
EXPRESSED IN TERMS OF CONCENTRATION IN AIR (ng/m^3)
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
Modified Method 5 Train Catches												
8/28/84		[45.95]	6.49	0.88	0.21	0.93	1.51	[81.22]	[12.95]	[2.47]	0.26	0.06
8/30/84		43.75	1.94	0.37	0.84	2.52	1.67	76.98	4.28	1.95	0.55	0.17
9/5/84		4.92				0.47		94.53	0.17			

NOTES

○ - Data out of control with respect to precision criteria (±50% RPD)

[ ] - Bracketed data denote homologues detected in filter and probe wash portion of Modified Method 5 train were deleted owing to unacceptable duplicate analysis results. Only a small fraction of total concentration detected was affected (see data in Table D-38).

- Matrix spike analyses indicated recoveries out of control for the following
  - Filter and probe wash - PeCDD and HxCDF
  - XAD-2 cartridge - HpCDD and HpCDF

Other media in the sampling train showed acceptable matrix spike recoveries.

TABLE D-40

INCINERATOR EXHAUST - PCDD/PCDF ANALYSES
FROM MODIFIED METHOD 5 TRAINS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
8/28/84												
Filter + Probe Wash	ND (0.638)	10.2	ND (0.160)	ND (0.301)	ND (0.431)	2.80	0.584	29.2	3.95	1.09	ND (0.428)	ND (0.592)
Field Blank	ND (0.107)	ND (0.111)	ND (0.276)	ND (0.191)	ND (1.27)	ND (5.49)	ND (0.138)	ND (0.186)	ND (0.154)	ND (0.223)	ND (0.662)	ND (3.56)
XAD-2 Cartridge	ND (3.63)	283	42.5	5.75	1.36	1.33	9.32	213	84.8	16.2	1.70	0.400
Field Blank	ND (.0630)	ND (.0209)	ND (0.143)	ND (0.119)	ND (0.127)	0.341	ND (0.297)	1.23	ND (0.357)	ND (0.470)	ND (.0707)	ND (0.122)
Impingers	ND (0.436)	16.6	ND (0.430)	ND (0.470)	ND (0.709)	2.29	ND (0.463)	287	ND (0.680)	ND (0.469)	ND (0.968)	ND (0.830)
Field Blank	ND (0.236)	ND (0.162)	ND (0.613)	ND (0.485)	ND (0.898)	2.19	ND (0.180)	ND (0.169)	ND (0.471)	ND (0.364)	ND (0.495)	ND (0.694)
Backup XAD-2	ND (0.135)	1.35	ND (0.146)	ND (.0768)	ND (.0605)	ND (0.441)	ND (0.481)	32.0	ND (.0778)	ND (.0742)	ND (0.112)	ND (0.105)
Field Blank	ND (.0862)	ND (.0542)	ND (0.340)	ND (0.180)	ND (0.216)	ND (0.338)	ND (.0704)	ND (.0649)	ND (0.148)	ND (0.130)	ND (0.301)	ND (0.326)
8/30/84												
Filter + Probe Wash	ND (0.597)	19.0	2.96	1.22	3.52	11.4	0.532	264	6.23	2.79	1.76	0.967
Field Blank	ND (.0736)	ND (.0150)	ND (.0558)	ND (.0425)	ND 0.119)	ND (0.271)	ND (.0433)	ND (.0171)	ND (.0623)	ND (.0663)	ND (.0833)	ND (0.218)
XAD-2 Cartridge	ND (16.6)	206	8.10	ND (0.339)	ND (0.358)	1.46	8.26	33.8	11.8	3.23	ND (0.360)	ND (0.646)
Field Blank	ND (0.132)	ND (0.154)	ND (0.421)	ND (0.411)	ND (0.345)	ND (0.369)	ND (0.170)	ND (0.175)	ND (0.230)	ND (0.333)	ND (0.345)	ND (0.368)
Impingers	ND (0.374)	24.4	ND (0.274)	0.878	1.26	1.52	0.712	141	6.34	5.11	1.39	ND (0.207)
Field Blank	ND (0.137)	ND (0.280)	ND (1.30)	ND (0.790)	ND (5.02)	ND (3.81)	ND (0.259)	ND (0.302)	ND (0.661)	ND (1.12)	ND (1.57)	ND (2.39)
Backup XAD-2												
Field Blank												
9/5/84												
Filter + Probe Wash	ND (0.324)	10.2	ND (0.160)	ND (0.109)	ND (0.443)	4.30	ND (0.379)	191	0.967	ND (0.124)	ND (0.771)	ND (0.220)
Field Blank	ND (0.109)	0.369	ND (.0769)	ND (.0483)	ND (0.129)	1.65	ND (.0264)	5.98	ND (.0555)	ND (.0939)	ND (0.132)	ND (0.125)
XAD-2 Cartridge	ND (0.198)	15.9	ND (.0787)	ND (.0616)	ND (0.371)	ND (0.281)	ND (0.322)	313	ND (.0734)	ND (.0566)	ND (0.340)	ND (0.556)
Field Blank	ND (0.801)	ND (1.04)	ND (1.86)	ND (1.74)	ND (2.73)	ND (2.69)	ND (0.847)	ND (0.905)	ND (1.28)	ND (1.93)	ND (3.24)	ND (2.00)
Impingers	ND (0.442)	2.11	ND (4.60)	ND (3.90)	ND (278)	ND (16.2)	ND (0.488)	36.1	ND (0.823)	ND (2.54)	ND (3.82)	ND (25.0)
Field Blank	ND (0.153)	ND (0.111)	ND (0.558)	ND (0.447)	ND (0.753)	ND (1.89)	ND (0.138)	ND (0.173)	ND (0.413)	ND (0.456)	ND (0.819)	ND (0.621)
Backup XAD-2												
Field Blank												

NOTES: Data expressed in nanograms per total sample. Accuracy (surrogate recovery) data appear in Table D-38.

TABLE D-41

INCINERATOR EXHAUST - TCDD ISOMERS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
8/28/84															
Filter + Probe Wash	0.69	3.74								5.29					
Field Blank															
XAD-2 Cartridge	122	75.9		11.0						73.5					
Field Blank															
Impingers	8.94	4.51								3.16					
Field Blank*															
Backup XAD-2	0.88	0.37								0.10					
Field Blank															
8/30/84															
Filter + Probe Wash	8.70	4.96		0.52						4.79					
Field Blank															
XAD-2 Cartridge	74.8	60.6								64.3					
Field Blank															
Impingers	12.3	6.65		0.62						4.81					
Field Blank															
Backup XAD-2*															
Field Blank*															
9/5/84															
Filter + Probe Wash	4.51	2.82		0.32						2.50					
Field Blank	0.81	0.07								0.12					
XAD-2 Cartridge	8.67	4.68		0.52						1.99					
Field Blank															
Impingers	2.11														
Field Blank															
Backup XAD-2*															
Field Blank*															

NOTE: Data expressed in nanograms per total sample.

* Sample analysis not returned from laboratory

TABLE D-42

INCINERATOR EXHAUST - TCDD ISOMERS
EXPRESSED IN TERMS OF CONCENTRATION IN AIR

DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
Notified Method 5 Train Catches															
8/28/84	20	13		1.8						12.5					
8/30/84	17	13		0.2						13					
9/5/84	2.9	1.3		0.2						0.8					

NOTE - Data expressed in ng/m^3

TABLE D-43

INCINERATOR EXHAUST - TCDD ISOMERS
AS MEASURED USING MODIFIED METHOD 5 TRAINS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, AND 9/5/84

(All data expressed in nanograms per total sample.)

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
8/28/84															
Filter + Probe Wash	0.694	3.74	ND (0.258)	0.980	ND (0.258)	ND (0.258)	ND (0.258)	ND (0.258)	ND (0.258)	5.29	ND (0.638)	ND (0.258)	ND (0.258)	ND (.0258)	ND (0.258)
Field Blank	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND 0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.107)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)
XAD-2 Cartridge	122	75.9	ND (3.09)	11.0	ND (3.09)	ND (3.09)	ND (3.09)	ND (3.09)	ND (3.09)	73.5	ND (3.63)	ND (3.09)	ND (6.17)	ND (3.09)	ND (6.17)
Field Blank	ND (.0209)	ND (.0209)	ND (.0209)	ND (.0209)	ND (.0209)	ND (.0209)	ND (.0209)	ND (.0290)	ND (.0290)	ND (.0290)	ND (.0630)	ND (.0290)	DN (.0290)	ND (.0290)	ND (.0290)
Impingers	8.94	4.51	ND (0.434)	ND (0.651)	ND (0.434)	ND (0.434)	ND (0.434)	ND (0.434)	ND (0.651)	3.16	ND (0.436)	ND (0.434)	ND (0.434)	ND (0.434)	ND (0.434)
Field Blank	ND (0.162)	ND (0.162)	ND (0.162)	ND (0.162)	ND (0.162)	ND (0.162)	ND (0.162)	ND (0.162)	ND (0.162)	ND (0.288)	ND (0.236)	ND (0.162)	ND (0.162)	ND (0.162)	ND (0.162)
Backup XAD-2	0.879	0.372	ND (.0431)	ND (.0647)	ND (.0647)	ND (0.108)	ND (.0518)	ND (.0431)	ND (0.431)	0.0997	ND (0.135)	ND (.0431)	ND (.0431)	ND (.0431)	ND (.0431)
Field Blank	ND (.0867)	ND (.0542)	ND (.136)	ND (.0542)	ND (.0542)	ND (.0542)	ND (.0542)	ND (.0542)	ND (.0542)	ND (.0542)	ND (.0862)	ND (.0542)	ND (.0542)	ND (.0542)	ND (.0542)
8/30/84															
Filter + Probe Wash	8.70	4.96	ND (0.217)	0.522	ND (0.217)	ND (0.217)	ND (0.217)	ND (0.217)	ND (0.217)	4.79	ND (0.597)	ND (0.217)	ND (0.217)	ND (0.217)	ND (0.217)
Field Blank	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0736)	ND (.0150)	ND (.0150)	ND (.0150)	ND (.0150)
XAD-2 Cartridge	74.8	60.6	ND (3.78)	ND (3.78)	ND (3.78)	ND (3.78)	ND (3.78)	ND (3.78)	ND (3.78)	64.3	ND (16.6)	ND (3.78)	ND (3.78)	ND (3.78)	ND (3.78)
Field Blank	ND (0.165)	ND (0.154)	ND (.154)	ND (0.154)	ND (0.154)	ND (0.154)	ND (0.154)	ND (0.154)	ND (0.154)	ND (0.154)	ND (0.132)	ND (0.154)	ND (0.154)	ND (0.154)	ND (0.154)
Impingers	12.3	6.65	ND (0.307)	0.616	ND (0.614)	ND (0.307)	ND (0.307)	ND (0.307)	ND (0.307)	4.81	ND (0.374)	ND (0.307)	ND (0.307)	ND (0.307)	ND (0.307)
Field Blank	ND (0.280)	ND (0.280)	ND (0.280)	ND (0.280)	ND (0.280)	ND (0.280)	ND (0.280)	ND (0.280)	ND (0.280)	ND (0.560)	ND (0.137)	ND (0.350)	ND (0.350)	ND (0.350)	ND (0.350)
Backup XAD-2	(Sample analysis not returned from laboratory.)														
Field Blank	(Sample analysis not returned from laboratory.)														
9/5/84															
Filter + Probe Wash	4.51	2.82	ND (0.231)	0.319	ND (0.231)	ND (0.231)	ND (0.231)	ND (0.231)	ND (0.231)	2.50	ND (0.324)	ND (0.231)	ND (0.231)	ND (0.231)	ND (0.231)
Field Blank	0.176	0.0732	ND (.0272)	ND (.0272)	ND (.0272)	ND (.0272)	ND (0.127)	ND (0.272)	ND (.0363)	0.121	ND (0.109)	ND (0.272)	ND (0.272)	ND (0.272)	ND (0.272)
XAD-2 Cartridge	8.67	4.68	ND (0.428)	0.520	ND (0.428)	ND (0.428)	ND (0.428)	ND (0.428)	ND (0.428)	1.99	ND (0.198)	ND (0.428)	ND (0.428)	ND (0.428)	ND (0.428)
Field Blank	ND (1.39)	ND (1.04)	ND (1.04)	ND (1.04)	ND (1.04)	ND (1.04)	ND (1.04)	ND (1.04)	ND (1.04)	ND (1.04)	ND (0.801)	ND (1.04)	ND (1.04)	ND (1.04)	ND (1.04)
Impingers	2.11	ND (0.581)	ND (0.581)	ND (0.581)	ND (0.581)	ND (0.581)	ND (0.581)	ND (0.581)	ND (0.581)	ND (0.697)	ND (0.442)	ND (0.581)	ND (0.581)	ND (0.581)	ND (1.16)
Field Blank	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.153)	ND (0.111)	ND (0.111)	ND (0.111)	ND (0.111)
Backup XAD-2	(Sample analysis not returned from laboratory.)														
Field Blank	(Sample analysis not returned from laboratory.)														

TABLE D-44

RESULTS OF SAMPLING FOR VINYLIDENE CHLORIDE
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR

DATE	SAMPLE RUN	SAMPLE COLLECTION TIME (EDT)	VINYLIDENE CHLORIDE CONCENTRATION (ppbv)	STANDARD DEVIATION
8/28/84	1	1230-1330	88.6 (83.1, 88.0, 94.7)	5.8
	2	1405-1510	68.3 (72.1, 72.3, 60.2)	6.9
	3	1525-1625	64.3 (113.0*, 67.5, 61.1)	4.5
	4	1640-1735	74.5 (73.9, 74.7, 74.8)	0.5
	5	1750-1845	88.9 (94.2, 88.4, 84.1)	5.1
	6	1850-1930	112.4 (113.6, 111.2, 138.6*)	1.7
	7	1935-2015	104.4 (102.1, 107.8, 103.3)	3.0
8/30/84	1	1000-1050	149.7 (150.0, 154.9, 144.3)	5.3
	2	1100-1200	187.6 (180.9, 189.3, 192.7)	6.1
	3	1210-1250	241.6 (263.7, 219.5, 402.7*)	31.3
	4	1300-1350	279.8 (275.3, 285.9, 278.3)	5.5
	5	1400-1450	218.0 (219.6, 216.3)	2.3
	6	1500-1550	28.1 (28.9, 27.9, 27.6)	0.7
9/5/84	1	1000-1045	88.7 (94.3, 93.3, 78.5)	8.8
	2	1100-1150	70.3 (69.4, 68.9, 72.6)	2.0
	2 DUPLICATE	1100-1150	79.3 (76.7, 81.9, 79.3)	2.6
	3	1200-1245	157.8 (156.4, 152.5, 164.4)	6.1
	4	1400-1445	154.3 (162.2, 143.5, 157.2)	9.7
	5	1500-1545	156.0 (154.7, 161.6, 151.8)	5.0
	6	1600-1630	143.5 (146.6, 143.3, 140.6)	3.0

* Rejected as greater than one standard deviation from mean of three analyses.

TABLE D-45

RESULTS OF SAMPLE AND BAG STABILITY TESTS
FOR VINYLIDENE CHLORIDE SAMPLES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28 AND 9/5/84

DATE	SAMPLE RUN	COMPARATIVE CONCENTRATION		DIFFERENCE (%)
		ANALYSIS DAY	FOLLOWING DAY	
8/28/84	5	88.9	63.5 (47.6*, 65.6, 61.4)	-28.6
	6	112.4	104.7 (108.4, 102.8, 103.0)	-6.9
	7	104.4	105.4 (112.2, 100.9, 103.2)	+1.0
9/5/84	1	88.7	84.8 (single value only)	-4.4
	5	156.0	178.4 (182.5, 183.9, 168.8)	+14.4
	6	143.5	179.1 (171.1, 183.2, 183.1)	+24.8

* Rejected as greater than one standard deviation from mean of three analyses.

E. Incinerator Ash

1. Semi-Volatiles

Analyses of incinerator ash (see Table D-46) revealed the presence of 1,2- and 1,4-dichlorobenzene; 1,2,4-trichlorobenzene; phenol; and biphenyl, among the targeted compounds. However, the first three compounds were found only in the field duplicate sample on the second sampling day, in the low ppm range. Phenol and biphenyl were detected at the 0.5 ppm level on the third sampling day. Tentatively identified in the ash collected on the second sampling day, in the sample and field duplicate, were the following ring compounds:

Table D-47

Semi-Volatile Compounds
Incinerator Ash
8/30/84

Compound	Concentration (mg/kg) Sample	Concentration (mg/kg) Field Duplicate	Precision (RPD)
Methyldiphenylsilane	52.838	44.757	16.6
1,1'-(1,2-ethendiyl)bis(z)benzene	11.628	5.661	69.0
1,1':2',1-terphenyl	4.932	9.919	67.2
1,1':3',1-terphenyl	10.792	6.245	53.4
1,1':4',1-terphenyl	11.243	9.965	12.1

Quality assurance criteria with respect to accuracy (surrogate recovery) were met for all of the samples analyzed. However, two of the seven samples, field blanks for the second and third sampling days, were lost prior to analysis by the laboratory.

2. PCDD/PCDF

a. All Homologues

These data, presented in Table D-48, appear to indicate that among the PCDDs, the homologues were detected in concentrations increasing according to their chlorine substitution; OCDD was most common. With PCDF, this relationship did not hold; total TCDF were generally most prevalent, followed by OCDF and hepta-CDF.

Accuracy criteria for the four surrogate compounds were not met for two of the six completed analyses; the seventh analysis was not accomplished, resulting in 57% completeness for the incinerator ash PCDD/PCDF analyses. Note, however, that the surrogate recovery criteria for $^{13}C_{12}$2378-TCDD and $^{37}Cl_{4}$2378-TCDF (70-130%) were missed by only 5 to 6%. The precision goal of ±50% was achieved for most homologues detected in the field duplicate samples obtained on the

TABLE D-46

INCINERATOR ASH SEMI-VOLATILES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
(Results in µg/kg)

																					ACCURACY (% SURROGATE RECOVERY)						
	1,2-dichlorobenzene	1,4-dichlorobenzene	1,2,4-trichlorobenzene	phenol	di-n-butyl phthalate	diethyl phthalate	bis (2-ethylhexyl) phthalate	1,1,1-oxy-bis-benzene	sulfur	biphenyl	2-ethylbiphenyl	methyldiphenylsilane	1,1'-Ω,2-ethendiyl)bis(z) benzene	1,1':2',1-terphenyl	1,1':3',1-terphenyl	1,1':4',1-terphenyl	octamethyltrisiloxane	2-phenoxy-1,1'-biphenyl	methylbiphenylsilane	bis (2-methylpropyl) phthalate	nitrobenzene-D5	2-fluorobiphenyl	terphenyl-D14	phenol-D5	2-fluorophenol	2,4,6-tribromophenol	Acceptable[1]
8/28/84					433			201	2722												102	135	67	101	86	35	Yes
8/28/84 field blank																					65	52	46	22	26	59	Yes
8/30/84					1933						7183	52838	11628	4932	10792	11243					96	117	83	44	59	136	Yes
8/30/84 field dup.	520	460	867		1733			10883				44757	5661	9919	6845	9965	2006	3681			30	35	72	63	92	110	Yes
8/30/84 field blank	(SAMPLE ANALYSIS NOT RETURNED FROM LABORATORY)																										
9/5/84				363	1110	423	530		170	435		321								1069	63	56	86	72	94	23	Yes
9/5/84 field blank	(SAMPLE ANALYSIS NOT RETURNED FROM LABORATORY)																										

Completeness = 5/7 = 71%

[1] All surrogate recoveries within target ranges established in Quality Assurance Project Plan

Note that recoveries of all base-neutral and acid surrogates were within the target range of 20 to 180%.

TABLE D-48

INCINERATOR ASH - PCDD/PCDF ANALYSES[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, AND 9/5/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	Accuracy (%Surrogate Recovery)			
													$^{13}C_{12}$2378-TCDD	$^{37}Cl_4$2378-TCDD	$^{13}C_{12}$OCDD	$^{37}Cl_4$2378-TCDF
8/28/84																
COMPOSITE SAMPLE	ND (27.7)	1170	ND (19.1)	793	6060	32,700	66	9160	68	455	1520	2570	88	97	100	80
FIELD BLANK	ND (8.2)	ND (9.6)	ND (35.8)	ND (17.5)	ND (12.7)	ND (25.8)	ND (12.6)	ND (12.8)	ND (21.2)	ND (19.6)	ND (15.9)	ND (23.4)	74	91	100	70
8/30/84																
COMPOSITE SAMPLE	ND (23.1)	131	ND (13.6)	129	806	3180	17	594	ND (5.4)	44	449	573	65	92	100	73
FIELD DUPLICATE	ND (11.8)	107	ND (15.6)	111	498	2370	ND (11.3)	263	ND (7.3)	37	248	399	64	95	100	65
PRECISION (RPD)		20		15	47	29		77		17	58	36				
FIELD BLANK													100	97	78	93
9/5/84																
COMPOSITE SAMPLE	ND (6.9)	71	ND (16.2)	ND (10.9)	76	266	ND (6.5)	540	ND (7.8)	ND (19.5)	ND (20.2)	78	84	98	100	78
FIELD BLANK		(Analytical data not returned from laboratory.)														
									COMPLETENESS BY SURROGATE				86%	86%	86%	86%

NOTES: [1] Data expressed in pg/g.
[2] All surrogate recoveries within target range of 50-150%.

second day. Detection limit goals of 5 ppt for TCDD and TCDF, and 15 ppt for other homologues, were generally met; detection limits of 0.5 to 1.9 ppt were achieved for TCDD and TCDF, and about 0.3 to 2.0 ppt for higher homologue groups.

b. TCDD Isomers

The 1368, 1379, and 1237/1238 isomers appeared in all samples; no 2378 isomer was found. Duplicate samples from the second day yielded satisfactory results for precision (see Table D-49).

F. Aqueous Influents and Effluents

This category of samples refers to those water streams circulated on a once-through basis in air pollution control equipment associated with the Building 703 incinerator, and the returned treated wastewaters ("service water") used to make up the bulk of water supplied to these devices (except the ESP, see Section V.A.2.d, and the ash pit, see Section V.A.3 of this report).

1. Volatile Compounds

Owing to the small volume of water samples taken (40 mL for volatiles compared to one gallon for semi-volatiles) and the correspondingly small fraction of solid matter in these samples, data with respect to volatile compounds in influent and effluent waters were reported in terms of micrograms per liter. Quality assurance criteria for accuracy (percent recovery of surrogates) were met for all but two of the 22 samples analyzed in this category; however, the analytical procedures did not achieve the target detection limit of 1 ppb, as the detection limit for most of the compounds of interest was 5 ppb.

The behavior of volatile compounds in air pollution control equipment waters appeared similar to that of the semi-volatiles discussed previously. That is, many compounds present in influent (service) water appeared to have been volatilized in contact with scrubbed exhaust gases from the incinerator. This phenomenon (see Tables D-50, D-51, and D-52) was observed in the cases of chloroform, carbon tetrachloride, and some other compounds.

The data revealed the regular presence of no distinct compounds. However, on the second sampling day, a number of compounds were found in ash pit effluent, among them the target compounds perchloroethylene and monochlorobenzene, and other constituents of interest, such as chloroethane, chloromethane, and ethylbenzene. This corresponds to a previously described finding of several semi-volatile compounds in ash pit solid matter on the second sampling day.

Field duplicate samples were obtained only of the ESP water stream on the second sampling day. Analytical results could be compared only for methylene chloride and dimethoxymethane; precision appeared good for the former but poor for the latter.

TABLE D-49

INCINERATOR ASH - TCDD ISOMERS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28, 8/30, AND 9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
8/28/84															
COMPOSITE SAMPLE	620	248		37						267					
FIELD BLANK															
8/30/84															
COMPOSITE SAMPLE	65	35		8						23					
FIELD DUPLICATE	57	31								19					
PRECISION (RPD)	13	12		--						19					
FIELD BLANK															
9/5/84															
COMPOSITE SAMPLE	35	23								14					
FIELD BLANK		(Analytical data not returned from laboratory.)													

NOTE: Data expressed in pg/g.

TABLE D-50

AQUEOUS INFLUENTS AND EFFLUENTS - VOLATILE COMPOUNDS[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

	TARGET COMPOUNDS				OTHER VOLATILE COMPOUNDS											TENTATIVELY IDENTIFIED COMPOUNDS						ACCURACY (% SURROGATE RECOVERY)			
SAMPLE IDENTIFICATION	chloroform	carbon tetrachloride	perchloroethylene	monochlorobenzene	methylene chloride	acetone	1,1,1-trichloroethane	1,2-dichloropropane	benzene	chloromethane	chloroethene	2-butanone	ethylbenzene	total xylenes	toluene	dimethoxymethane	1,2,3-trichloropropane	cyclohexane	2,3-dimethylbutane	hexamethylcyclotrisiloxane	hexane	toluene-D8	bromofluorobenzene	1,2-dichloroethane-D4	ACCEPTABLE[2]
INFLUENT																									
SERVICE WATER	4*	16			41			6								289						111	91	84	YES
EFFLUENTS																									
QUENCH WATER																						111	99	103	YES
VENTURI/DEMISTER WATER																73						104	103	91	YES
ESP WATER																173						104	111	97	YES
ESP WATER FIELD DUPLICATE	(No sample taken.)																								---
ASH PIT WATER					6																	103	103	102	YES
FIELD BLANKS																									
EFFLUENT WATER					232																	102	117	103	YES
EFFLUENT WATER DUPLICATE					169																	102	111	103	YES

Note - [1] Data expressed in ug/L.
[2] All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.

* Estimated value.

Completeness =
7/7 = 100%

TABLE D-51

AQUEOUS INFLUENTS AND EFFLUENTS - VOLATILE COMPOUNDS[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

SAMPLE IDENTIFICATION	TARGET COMPOUNDS				OTHER VOLATILE COMPOUNDS											TENTATIVELY IDENTIFIED COMPOUNDS						ACCURACY (% SURROGATE RECOVERY)			ACCEPTABLE[2]
	chloroform	carbon tetrachloride	perchloroethylene	monochlorobenzene	methylene chloride	acetone	1,1,1-trichloroethane	1,2-dichloropropane	benzene	chloromethane	chloroethene	2-butanone	ethylbenzene	total xylenes	toluene	dimethoxymethane	1,2,3-trichloropropane	cyclohexane	2,3-dimethylbutane	hexamethylcyclotrisiloxane	hexane	toluene-D8	bromofluorobenzene	1,2-dichloroethane-D4	
INFLUENT																									
SERVICE WATER	7	71			68		9										17					96	80	84	YES
EFFLUENTS																									
QUENCH WATER					5																	90	104	104	YES
VENTURI/DEMISTER WATER					8	19																93	88	108	YES
ESP WATER					4*											3						89	94	96	YES
ESP WATER FIELD DUPLICATE																						94	99	105	YES
ASH PIT WATER			218		5	30				94	37	41	96	117	184							97	95	106	YES
FIELD BLANKS																									
EFFLUENT WATER					20	33			11												33	94	68	106	NO
EFFLUENT WATER DUPLICATE					19	15			7												32	91	84	112	YES

Note - [1] Data expressed in ug/L.
[2] All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.

* Estimated value.

Completeness = 7/8 = 88%

TABLE D-52

AQUEOUS INFLUENTS AND EFFLUENTS - VOLATILE COMPOUNDS[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

	TARGET COMPOUNDS				OTHER VOLATILE COMPOUNDS											TENTATIVELY IDENTIFIED COMPOUNDS						ACCURACY (% SURROGATE RECOVERY)			
SAMPLE IDENTIFICATION	chloroform	carbon tetrachloride	perchloroethylene	monochlorobenzene	methylene chloride	acetone	1,1,1-trichloroethane	1,2-dichloropropane	benzene	chloromethane	chloroethene	2-butanone	ethylbenzene	total xylenes	toluene	dimethoxymethane	1,2,3-trichloropropane	cyclohexane	2,3-dimethylbutane	hexamethylcyclotrisiloxane	hexane	toluene-D8	bromofluorobenzene	1,2-dichloroethane-D4	ACCEPTABLE[2]
INFLUENT																									
SERVICE WATER	21	110	4		55		6	16	4													97	99	80	YES
EFFLUENTS																									
QUENCH WATER					24				5											200		99	100	91	YES
VENTURI/DEMISTER WATER					2600	1198			5					5						200		99	107	92	YES
ESP WATER					16				7							225						99	98	81	YES
ESP WATER FIELD DUPLICATE	(No sample taken.)																								
ASH PIT WATER									4													98	118	96	YES
FIELD BLANKS																									
EFFLUENT WATER					70	51	5								1.6*							93	108	115	YES
EFFLUENT WATER DUPLICATE					26	44			4													101	91	96	YES

Note - [1]Data expressed in ug/L.
[2]All surrogate recoveries within target range (80-125%) established in Quality Assurance Project Plan.

* Estimated value.

Completeness = 7/7 = 100%

2. Semi-Volatile Compounds

Quality assurance criteria with respect to accuracy (surrogate recoveries) were generally met for the analyses of aqueous samples and solid filtrates. However, detection limit goals of 5 ppb in liquids and solids were not achieved, actual detection limits being on the order of 10 ppb. The analytical data, Tables D-53, D-54, and D-55, show that few semi-volatile compounds were detected in any of the wastewater liquid and solid streams at levels higher than those in influent service waters. This appears to indicate that such compounds present in influent waters may have volatilized out of the liquids as they passed through the incinerator air pollution control devices.

Effluent waters were found to contain only the following targeted compounds on the sampling days indicated:

Compound	Sampling Day	Concentration (ug/L)	Effluent Stream
Tetrachlorophenol	2	13	Venturi/Demister
Monochlorobenzene	2	157	Ash Pit
1,1'-biphenyl	2	285	Ash Pit

However, surrogate recoveries from the sample from which the last two compounds were analyzed did not meet the quality assurance goals established for the study, and these data should therefore be considered tentative.

Little was detected in the filtered solids portions of the effluent streams. As the data in Tables D-53, D-54, and D-55 indicate, several phthalates were found regularly, and many effluent streams contained solid elemental sulfur. Of possible interest is the finding of biphenyls in electrostatic precipitator and ash pit effluent solids on the third sampling day, and a variety of benzene, biphenyl, and terphenyl compounds in ash pit effluent solids on the second sampling day. Note that several of these compounds also appeared in incinerator ash on that day.

3. PCDD/PCDF

a. All Homologues

It will be recalled from previous descriptions of the Dow facility that service water circulated in most incinerator air pollution control devices is composed of a stream of wastewater from the plant's wastewater treatment system. Low concentrations of tetra- and octa-CDD and CDF were detected on the first and second sampling days, along with traces of other homologues on the latter day; no PCDD or PCDF were found on the third day (see Tables D-56, D-57, and D-58).

Effluent water stream concentrations of all homologues appear several orders of magnitude higher than in service water. However, note that PCDD and PCDF reside almost exclusively in the suspended (filterable) solids present in these

TABLE D-53

AQUEOUS INFLUENTS AND EFFLUENTS - SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

SAMPLE IDENTIFICATION	UNITS	TARGET COMPOUNDS										OTHER COMPOUNDS																										Accuracy (% Surrogate Recovery)						ACCEPTABLE[1]
																																						Base-Neutrals			Acids			
		2,4,6-trichlorophenol*	tetrachlorophenol	2,3,4,6-tetrachlorophenol	pentachlorophenol*	biphenyl	1,1'-biphenyl	monochlorobenzene	1,2-dichlorobenzene*	1,2,4-trichlorobenzene*	1,2,4,5-tetrachlorobenzene	1-ethyl-2-methylbenzene	di-n-butyl phthalate*	diethyl phthalate	bis(2-ethylhexyl)phthalate*	di-n-octyl phthalate*	bis(2-chloroethyl) ether*	benzoic acid*	hexadecanoic acid	sulfur	bis(2-methylpropyl) phthalate	butyl-2-methylpropyl phthalate	1,1'-biphenyl-4-ol	2-ethyl-1,1'-biphenyl	methyldiphenylsilane	1,1'-(1,2-ethendiyl)bis-(z)-benzene	1,1':2',1-terphenyl	1,1':3',1-terphenyl	1,1-diphenylheptane	diphenylether	butyloctadecanoate	2,2'-oxybisethanol	1,3-dimethylbenzene	1-butoxy-2-propanol	2-(2-butoxyethoxy) ethanol	1,1,3-trimethylcyclopentane	1-(2-butoxyethoxy) ethanol	nitrobenzene-D5	2-fluorobiphenyl	terphenyl-D14	phenol-D5	2-fluorophenol	2,4,6-tribromophenol	
Service Water	ug/L																		28																			57	60	77	69	51	54	YES
Quench Water (Water Portion)	ug/L																																					72	59	91	37	35	67	YES
Quench Water (Solids Portion)	ug/kg												18							35																		57	67	98	37	73	71	YES
Venturi/Demister Water (Water Portion)	ug/kg																																					72	63	81	82	84	68	YES
Venturi/Demister Water (Solids Portion)	ug/L												21							32																		24	30	44	22	26	32	YES
ESP Water (Water Portion)	ug/L																																					83	70	95	26	101	90	YES
ESP Water Water Field Duplicate	ug/L		(No sample taken.)																																									
ESP Water (Solids Portion)	ug/kg												300	132							336																	72	104	46	43	37	67	YES
ESP Water Solids Field Duplicate	ug/kg		(No sample taken.)																																									
Ash Pit Water (Water Portion)	ug/L																																					54	48	58	29	61	51	YES
Ash Pit Water (Solids Portion)	ug/kg												34,750		82,725				107,155	242,536	36,212																	32	38	122	31	38	52	YES
Effluent Water Field Blank	ug/L																																					61	66	96	21	39	103	YES
Effluent Water Backup Field Blank	ug/L																																					46	50	66	17	49	85	NO

	Base-Neutrals	Acids	Overall
COMPLETENESS[2]	100%	91%	91% (10/11)

NOTES - All compounds were tentatively identified unless indicated by an asterisk.

[1] All surrogate recoveries within target range (20-180%) established in Quality Assurance Project Plan.

[2] By class of surrogates (acids and base-neutrals) and overall (combined).

TABLE D-54

AQUEOUS INFLUENTS AND EFFLUENTS - SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

		TARGET COMPOUNDS										OTHER COMPOUNDS																										Accuracy (% Surrogate Recovery)						
																																						Base-Neutrals			Acids			
SAMPLE IDENTIFICATION	UNITS	2,4,6-trichlorophenol*	tetrachlorophenol	2,3,4,6-tetrachlorophenol	pentachlorophenol*	biphenyl	1,1'-biphenyl	monochlorobenzene	1,2-dichlorobenzene*	1,2,4-trichlorobenzene*	1,2,4,5-tetrachlorobenzene	1-ethyl-2-methylbenzene	di-n-butyl phthalate*	diethyl phthalate	bis(2-ethylhexyl)phthalate*	di-n-octyl phthalate*	bis(2-chloroethyl) ether*	benzoic acid*	hexadecanoic acid	sulfur	bis(2-methylpropyl) phthalate	butyl-2-methylpropyl phthalate	1,1'-biphenyl-4-ol	2-ethyl-1,1'-biphenyl	methyldiphenylsilane	1,1'-(1,2-ethenediyl)bis-(Z)-benzene	1,1':2',1-terphenyl	1,1':3',1-terphenyl	1,1-diphenylheptane	diphenylether	butyloctadecanoate	2,2'-oxybisethanol	1,3-dimethylbenzene	1-butoxy-2-propanol	2-(2-butoxyethoxy) ethanol	1,1,3-trimethylcyclopentane	1-(2-butoxyethoxy) ethanol	nitrobenzene-D5	2-fluorobiphenyl	terphenyl-D14	phenol-D5	2-fluorophenol	2,4,6-tribromophenol	ACCEPTABLE¹
Service Water	ug/L				82																																	41	44	103	35	50	22	YES
Quench Water (Water Portion)	ug/L														25								33														1378	63	59	76	49	72	42	YES
Quench Water (Solids Portion)	ug/kg												90,497			4524				1864		548	27,060															64	62	84	68	76	68	YES
Venturi/Demister Water (Water Portion)	ug/L	19	13												19		1.8																					63	58	39	67	86	36	YES
Venturi/Demister Water (Solids Portion)	ug/kg												43,624		15,436																	48						55	76	66	53	54	52	YES
ESP Water (Water Portion)	ug/L												66																									84	104	55	52	44	80	YES
ESP Water Water Field Duplicate	ug/L														12																						747	84	78	98	81	86	99	YES
ESP Water (Solids Portion)	ug/kg												112,359		59,988							115,423																66	95	64	84	92	63	YES
ESP Water Solids Field Duplicate	ug/kg												412,849		89,908																							73	69	89	57	30	82	YES
Ash Pit Water (Water Portion)	ug/L						285								34										12								20				1625	82	97	119	58	105	187	NO
Ash Pit Water (Solids Portion)	ug/kg						1,678,504					12,876	68,571		58,000				56,105	15,348				31,778	729,436	130,871	118,101	119,269	13,643									95	67	119	28	33	101	YES
Effluent Water Field Blank	ug/L																																					60	51	65	29	44	96	YES
Effluent Water Backup Field Blank	ug/L																																					60	50	88	37	52	113	YES

	Base-Neutrals	Acids	Overall
COMPLETENESS²	100%	92%	92% (12/13)

NOTES - All compounds were tentatively identified unless indicated by an asterisk.
¹All surrogate recoveries within target range (20-180%) established in Quality Assurance Project Plan.
²By class of surrogates (acids and base-neutrals) and overall (combined).

TABLE D-55

AQUEOUS INFLUENTS AND EFFLUENTS - SEMI-VOLATILE COMPOUNDS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

SAMPLE IDENTIFICATION	UNITS	TARGET COMPOUNDS										OTHER COMPOUNDS																										Accuracy (% Surrogate Recovery)						
																																						Base-Neutrals			Acids			
		2,4,6-trichlorophenol*	tetrachlorophenol	2,3,4,6-tetrachlorophenol	pentachlorophenol*	biphenyl	1,1'-biphenyl	monochlorobenzene	1,2-dichlorobenzene*	1,2,4-trichlorobenzene*	1,2,4,5-tetrachlorobenzene	1-ethyl-2-methylbenzene	di-n-butyl phthalate*	diethyl phthalate	bis(2-ethylhexyl)phthalate*	di-n-octyl phthalate*	bis(2-chloroethyl) ether*	benzoic acid*	hexadecanoic acid	sulfur	bis(2-methylpropyl) phthalate	butyl-2-methylpropyl phthalate	1,1'-biphenyl-4-ol	2-ethyl-1,1'-biphenyl	methyldiphenylsilane	1,1'-(1,2-ethenediyl)bis-(2)-benzene	1,1':2',1-terphenyl	1,1':3',1-terphenyl	1,1-diphenylheptane	diphenylether	butyloctadecanoate	2,2'-oxybisethanol	1,3-dimethylbenzene	1-butoxy-2-propanol	2-(2-butoxyethoxy) ethanol	1,1,3-trimethylcyclopentane	1-(2-butoxyethoxy) ethanol	nitrobenzene-D5	2-fluorobiphenyl	terphenyl-D14	phenol-D5	2-fluorophenol	2,4,6-tribromophenol	ACCEPTABLE¹
Service Water	ug/L	22.8		15					4.6	6.8	13				14.8			451																				98	67	118	72	89	111	YES
Quench Water (Water Portion)	ug/kg														44																							43	94	45	60	47	88	YES
Quench Water (Solids Portion)	ug/L												153,979		301,454					21,750	66,024										604,357			19,301				61	80	90	84	75	120	YES
Venturi/Demister Water (Water Portion)	ug/kg			9											97																						815	74	94	85	81	80	101	YES
Venturi/Demister Water (Solids Portion)	ug/L														78,048					17,313											350,364	77,031				15,008		85	62	112	57	43	87	YES
ESP Water (Water Portion)	ug/L		(Sample analysis not returned from laboratory.)																																									---
ESP Water Water Field Duplicate	ug/L		(No sample taken.)																																									
ESP Water (Solids Portion)	ug/kg					37,500							233,849		127,376	19,011														95,656	205,259							48	82	143	27	31	131	YES
ESP Water Solids Field Duplicate	ug/kg		(No sample taken.)																																									
Ash Pit Water (Water Portion)	ug/L		(Sample analysis not returned from laboratory.)																																									---
Ash Pit Water (Solids Portion)	ug/kg					94,564							32,484	14,968	17,356					653,650	112,517				22,197									57,680				42	80	47	53	37	92	YES
Effluent Water Field Blank	ug/L														56																							99	86	93	30	97	110	YES
Effluent Water Backup Field Blank	ug/L														21																							91	57	77	45	103	103	YES

	Base-Neutrals	Acids	Overall
COMPLETENESS²	82%	82%	82% (9/11)

NOTES - All compounds were tentatively identified unless indicated by an asterisk.
[1] All surrogate recoveries within target range (20-180%) established in Quality Assurance Project Plan.
[2] By class of surrogates (acids and base-neutrals) and overall (combined). Note that all surrogate recoveries were within range; completeness was compromised only because laboratory did not analyze two samples.

TABLE D-56

AQUEOUS INFLUENTS AND EFFLUENTS - PCDD/PCDF ANALYSES[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

													Accuracy (% Surrogate Recovery)			
SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	Total OCDF	$^{13}C_{12}$2378-TCDD	$^{37}Cl_4$2378-TCDD	$^{13}C_{12}$OCDD	$^{37}Cl_4$2378-TCDF
Service Water	ND (.0021)	0.0384	ND (.0043)	ND (.0086)	ND (.0073)	0.198	ND (.0011)	1.26	ND (.0026)	ND (.0057)	0.0558	ND (.0130)	100	91	63	81
Quench Water (Water)	ND (.0013)	ND (.0010)	ND (.0010)	ND (.0042)	ND (.0079)	ND (.0206)	ND (.0005)	0.0025	ND (.0015)	ND (.0029)	ND (.0055)	ND (.0118)	100	81	87	62
Quench Water (Solids)	ND (15.6)	432	54.9	43.7	274	1437	11.0	170	66.4	117	427	379	93	94	84	100
Venturi/Demister Water (Water)	ND (.0011)	ND (.0010)	ND (.0027)	ND (.0026)	ND (.0059)	ND (.0147)	ND (.0002)	0.0393	ND (.0022)	ND (.0018)	ND (.0030)	ND (.0139)	62	89	100	57
Venturi/Demister Water (Solids)	ND (2.98)	238	82.0	55.1	265	1113	8.52	137	100	130	337	284	47	95	100	49
ESP Water (Water)		(Sample analysis data not returned from laboratory.)														
ESP Water (Solids)		(Sample analysis data not returned from laboratory.)														
Ash Pit Water (Water)	ND (.0003)	ND (.0010)	ND (.0010)	ND (.0027)	ND (.0058)	ND (.0289)	ND (.0003)	ND (.0010)	ND (.0031)	ND (.0012)	ND (.0066)	ND (.0121)	100	90	98	59
Ash Pit Water (Solids)	ND (19.8)	ND (23.3)	ND (171)	ND (94.3)	ND (126)	323	ND (27.4)	189	ND (45.1)	ND (42.5)	ND (91.5)	ND (118)	46	95	100	55
Effluent Water Field Blank	ND (.0003)	ND (.0010)	ND (.0016)	ND (.0026)	ND (.0083)	ND (.0130)	ND (.0002)	ND (.0010)	ND (.0039)	ND (.0014)	ND (.0055)	ND (.0098)	100	84	43	62
Effluent Water Backup Field Blank	ND (.0002)	ND (.0010)	ND (.0054)	ND (.0115)	ND (.0275)	ND (.0447)	ND (.0003)	ND (.0010)	ND (.0037)	ND (.0075)	ND (.0167)	ND (.0284)	30	80	10	20
									COMPLETENESS BY SURROGATE				55%	82%	64%	64%

Notes - [1] Data expressed in ng/g.
[2] All surrogate recoveries within range of 50-150%.

TABLE D-57

AQUEOUS INFLUENTS AND EFFLUENTS - PCDD/PCDF ANALYSES[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

													Accuracy (% Surrogate Recovery)			
SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	$^{13}C_{12}$2378-TCDD	$^{37}Cl_4$2378-TCDD	$^{13}C_{12}$OCDD	$^{37}Cl_4$2378-TCDF
Service Water	ND (.0027)	0.0464	ND (.0019)	ND (.0021)	0.0179	0.187	ND (.0012)	1.42	0.0088	ND (.0067)	0.0167	0.0477	95	110	100	66
Quench Water (Water)	ND (.0007)	ND (.0010)	ND (.0024)	ND (.0042)	ND (.0115)	ND (.0301)	ND (.0001)	0.0223	ND (.0037)	ND (.0028)	ND (.0131)	ND (.0168)	100	80	95	57
Quench Water (Solids)	ND (11.1)	707	99.3	75.3	460	2358	15.4	182	ND 87.5	124	785	641	62	94	93	100
ESP Water (Water)	ND (.0009)	.0062	ND (.0011)	ND (.0028)	ND (.0057)	ND (.0192)	ND (.0004)	0.287	ND (.0051)	ND (.0037)	ND (.0055)	ND (.0182)	100	111	67	44
ESP Water (Water) Field Duplicate	ND (.0028)	.0189	ND (.0019)	ND (.0029)	ND (.0044)	ND (.0077)	ND (.0004)	0.607	ND (.0039)	ND (.0017)	ND (.0070)	ND (.0099)	100	99	89	73
ESP Water (Solids)	ND (35.3)	4212	885	147	417	2199	45.3	539	405	75.7	150	200	85	88	100	95
ESP Water (Solids) Field Duplicate	ND (65.5)	1864	393	205	515	2530	47.7	6574	345	58.6	161	226	28	99	100	27
Venturi/Demister Water (Water)	ND (.0006)	ND (.0010)	ND (.0012)	ND (.0021)	ND (.0089)	ND (.0075)	ND (.0005)	0.0682	ND (.0021)	ND (.0033)	ND (.0056)	ND (.0164)	100	102	68	46
Venturi/Demister Water (Solids)	ND (2.08)	307	49.2	27.6	162	707	3.22	168	64.6	82.9	199	283	63	89	100	57
Ash Pit Water (Water)	ND (.0010)	ND (.0025)	ND (.0240)	ND (.0227)	ND (.0292)	ND (.0453)	ND (.0022)	ND (.0038)	ND (.0120)	ND (.0110)	ND (.0232)	ND (.0269)	38	91	50	29
Ash Pit Water (Solids)	ND (1.08)	ND 15.9	ND (3.09)	ND (3.14)	21.5	94.9	ND (1.71)	114	ND (3.15)	ND (2.93)	10.0	12.5	73	94	100	65
Effluent Water Field Blank	ND (.0005)	ND (.0010)	ND (.0011)	ND (.0021)	ND (.0031)	ND (.0053)	ND (.0006)	ND (.0010)	ND (.0024)	ND (.0017)	ND (.0052)	ND (.0037)	92	90	55	36
Effluent Water Backup Field Blank	ND (.0005)	ND (.0010)	ND (.0080)	ND (.0063)	ND (.0083)	ND (.0104)	ND (.0014)	ND (.0025)	ND (.0077)	ND (.0128)	ND (.0046)	ND (.0127)	63	114	100	17
									COMPLETENESS BY SURROGATE				85%	100%	100%	54%

NOTES - [1] Data expressed in ng/g.
[2] All surrogate recoveries within range of 50-150%.

TABLE D-58

AQUEOUS INFLUENTS AND EFFLUENTS - PCDD/PCDF ANALYSES[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

													Accuracy (% Surrogate Recovery)			
SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	$^{13}C_{12}$2378-TCDD	$^{37}Cl_4$2378-TCDD	$^{13}C_{12}$OCDD	$^{37}Cl_4$2378-TCDF
Service Water	ND (0.341)	ND (0.229)	ND (0.556)	ND (0.720)	ND (0.318)	ND (0.520)	ND (0.192)	ND (0.517)	ND (0.299)	ND (0.351)	ND (0.627)	ND (0.396)	100	93	58	82
Quench Water (Water)	ND (.0004)	ND (.0010)	ND (.0024)	ND (.0027)	ND (.0018)	ND (.0020)	ND (.0001)	0.0058	ND (.0015)	ND (.0015)	ND (.0012)	ND (.0011)	53	107	100	30
Quench Water (Solids)	ND (1.10)	73.9	ND (7.43)	ND (3.19)	69.0	236	ND (1.93)	830	7.09	16.1	125	103	81	96	48	100
Venturi/Demister Water (Water)	ND (.0008)	ND (.0010)	ND (.0021)	ND (.0031)	ND (.0036)	ND (.0064)	ND (.0001)	0.0157	ND (.0010)	ND (.0024)	ND (.0017)	ND (.0035)	84	89	53	53
Venturi/Demister Water (Solids)	ND (1.29)	56.3	17.5	7.35	44.3	261	2.05	723	22.3	19.7	69.1	84.8	100	92	94	100
ESP Water (Water)	ND (.0014)	0.0052	ND (.0104)	ND (.0039)	ND (.0087)	ND (.0051)	ND (.0015)	0.0995	ND (.0041)	ND (.0030)	ND (.0026)	ND (.0061)	53	97	35	54
ESP Water (Solids)	ND (28.2)	247	61.5	20.3	96.0	423	9.70	90.0	47.0	14.7	68.2	82.1	70	91	39	100
Ash Pit Water (Water)	ND (.0003)	ND (.0010)	ND (.0012)	ND (.0017)	ND (.0029)	ND (.0025)	ND (.0001)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0021)	ND (.0037)	100	110	48	89
Ash Pit Water (Solids)		(Sample analysis data not returned from laboratory.)														
Effluent Water Field Blank	ND (.0013)	ND (.0010)	ND (.0016)	ND (.0071)	ND (.0067)	ND (.0088)	ND (.0023)	ND (.0022)	ND (.0080)	ND (.0025)	ND (.0049)	ND (.0057)	70	93	100	17
Effluent Water Backup Field Blank	ND (.0003)	ND (.0010)	ND (.0048)	ND (.0027)	ND (.0039)	ND (.0058)	ND (.0002)	ND (.0010)	ND (.0025)	ND (.0027)	ND (.0026)	ND (.0039)	48	94	100	26
									COMPLETENESS BY SURROGATE				82%	91%	55%	64%

NOTES - [1]Data expressed in ng/g.
[2]All surrogate recoveries within range of 50-150%.

once-through effluents. A full range of homologues was found on all three sampling days, though from these data it did not appear that PCDD and PCDF appear consistently in any particular wastewater stream.

No 2378-TCDD was detected on any day, but 2378-TCDF was found on three days. In general, the range and concentrations of all homologues was greater by one to two orders of magnitude on the second sampling day than on the other days. Particularly high concentrations of tetra- and octa-CDD and CDF were present in the solids fractions of the wastewaters.

Complete data sets covering all wastewater streams were not returned from the analytical laboratory for any but the second sampling day. Overall completeness, taking accuracy criteria into account, was 17% (6 of 35). Twenty-six of the 29 data sets were incomplete because of unsatisfactory accuracy. Field duplicate samples were taken only on the second day, of the ESP water stream. Calculations shown in Table D-59 reveal mixed precision between these sample data; generally good precision was achieved with higher homologues.

Detection limit criteria:

	2378- and Total Tetras	Penta- through Octa-
Waters	30 ppq	90 ppq
Solids	5 ppt	15 ppt,

were not met for water fraction analyses, with actual detection limits in the range of about 20 to 1600 ppq, nor for solids analyses, where detection limits were in the 0.6 to 6.0 ppb range (see Tables D-56, D-57, and D-58).

b. TCDD Isomers

As indicated previously, no 2378-TCDD was detected at any time in influent or effluent water streams. The data presented in Tables D-60, D-61, and D-62 indicate most TCDD appeared as the 1368, 1237/1238, and 1379 isomers. Occasionally, the 1369 isomer was observed, and on the second day, when the highest concentrations of PCDD/PCDF appeared, the 1247/1248/1378/1469 combination was noted.

Precision data obtained for the second day's samples are presented in Table D-63 and indicate generally poor performance in this area. The detection limit goals of 30 and 90 ppq, respectively, for water and solids fractions, were not achieved for the latter, with actual sensitivities one to two orders of magnitude lower.

TABLE D-59

AQUEOUS INFLUENT AND EFFLUENT WASTEWATER SAMPLE PRECISION
PCDD/PCDF HOMOLOGUES
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

SAMPLE IDENTIFICATION	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF
WATER FRACTION												
ESP Water		0.0062						0.287				
ESP Water Field Duplicate		0.0189						0.607				
Precision (RPD)		101						56				
FILTERABLE SOLIDS FRACTION												
ESP Water		4212	885	147	417	2199	45.3	539	405	75.7	150	200
ESP Water Field Duplicate		1864	393	205	515	2530	47.7	6574	345	58.6	161	226
Precision (RPD)		65	77	33	21	14	5	170	16	25	7	12

NOTE - Concentration data in ng/g.

TABLE D-60

AQUEOUS INFLUENTS AND EFFLUENTS - TCDD ISOMERS [1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
Service Water	0.0172	0.0095	ND (.0022)	ND (.0012)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	0.0100	ND (.0021)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Quench Water (Water)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0013)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Quench Water (Solids)	183	113	ND (4.57)	7.32	ND (4.57)	ND (4.57)	ND (9.14)	ND (4.57)	ND (4.57)	128	ND (15.6)	ND (4.57)	ND (4.57)	ND (4.57)	ND (4.57)
Venturi/Demister Water (Water)	ND (.0013)	ND (.0011)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0011)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Venturi/Demister Water (Solids)	113	ND (3.91)	7.83	ND (3.91)	ND (3.91)	ND (3.91)	ND (3.91)	ND (3.91)	ND (3.91)	117	ND (2.98)	ND (3.91)	ND (3.91)	ND (3.91)	ND (3.91)
ESP Water (Water)			(Sample analysis data not returned from laboratory)												
ESP Water (Solids)			(Sample analysis data not returned from laboratory)												
Ash Pit Water (Water)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0003)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Ash Pit Water (Solids)	ND (23.3)	ND (23.3)	ND (23.3)	ND (23.3)	ND (23.3)	ND (23.3)	ND (23.3)	ND (23.3)	ND (23.3)	ND (23.3)	ND (19.8)	ND (23.3)	ND (23.3)	ND (23.3)	ND (23.3)
Effluent Water Field Blank	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0003)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Effluent Water Backup Field Blank	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0002)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)

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[1] Data expressed in ng/g.

TABLE D-61

AQUEOUS INFLUENTS AND EFFLUENTS - TCDD ISOMERS[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
Service Water	0.0198	0.0154	ND (.0010)	ND (.0016)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	0.0093	ND (.0027)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Quench Water (Water)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0007)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Quench Water (Solids)	290	183	ND (7.23)	14.5	ND (7.23)	ND (7.23)	ND (7.23)	ND (7.23)	ND (7.23)	220	ND (11.1)	ND (7.23)	ND (7.23)	ND (7.23)	ND (7.23)
ESP Water (Water)	0.0038	0.0025	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0031)	ND (.0009)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
ESP Water (Water) Field Duplicate	0.0074	0.0057	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	0.0058	ND (.0028)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
ESP Water (Solids)	1968	945	ND (48.4)	59.0	ND (48.4)	ND (48.4)	ND (48.4)	ND (48.4)	ND (48.4)	1240	ND (35.3)	ND (48.4)	ND (48.4)	ND (48.4)	ND (48.4)
ESP Water (Solids) Field Duplicate	486	ND (32.8)	65.6	ND (32.8)	ND (32.8)	ND (32.8)	ND (32.8)	ND (32.8)	ND (32.8)	1313	ND (65.5)	ND (32.8)	ND (32.8)	ND (32.8)	ND (32.8)
Venturi/Demister Water (Water)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0006)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Venturi/Demister Water (Solids)	130	85.5	ND (3.23)	7.77	ND (3.23)	ND (3.23)	ND (3.23)	ND (3.23)	ND (3.23)	84.2	ND (2.08)	ND (3.23)	ND (3.23)	ND (3.23)	ND (3.23)
Ash Pit Water (Water)	ND (.0025)	ND (.0025)	ND (.0025)	ND (.0025)	ND (.0025)	ND (.0025)	ND (.0025)	ND (.0025)	ND (.0025)	ND (.0025)	ND (.0010)	ND (.0025)	ND (.0025)	ND (.0025)	*
Ash Pit Water (Solids)	74.7	3.97	ND (0.998)	0.934	ND (.998)	ND (.998)	ND (.998)	ND (.998)	ND (.998)	3.50	ND (1.08)	ND (0.998)	ND (0.998)	ND (0.998)	ND (1.20)
Effluent Water Field Blank	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0005)	ND (.0010)	ND (.0010)	ND (.0010)	*
Effluent Water Backup Field Blank	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0005)	ND (.0010)	ND (.0010)	ND (.0011)	*

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[1] Data expressed in ng/g.

* Denotes data not reported by laboratory.

TABLE D-62

AQUEOUS INFLUENTS AND EFFLUENTS - TCDD ISOMERS[1]
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

SAMPLE IDENTIFICATION	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
Service Water	ND (.0229)	ND (0.229)	ND (0.229)	ND (0.229)	ND (0.229)	ND (0.229)	ND (0.229)	ND (0.229)	ND (0.229)	ND (0.229)	ND (0.341)	ND (0.229)	ND (0.229)	ND (0.229)	ND (0.229)
Quench Water (Water)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0004)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Quench Water (Solids)	26.9	24.7	ND (1.31)	ND (2.62)	ND (1.97)	ND (1.97)	ND (1.97)	ND (1.97)	ND (1.97)	22.2	ND (1.10)	ND (1.97)	ND (1.97)	ND (1.97)	ND (1.97)
Venturi/Demister Water (Water)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0008)	ND (.0010)	ND (.0010)	ND (.0010)	*
Venturi/Demister Water (Solids)	28.2	ND (0.985)	2.01	ND (0.985)	ND (0.985)	ND (0.985)	ND (0.985)	ND (0.985)	ND (0.985)	26.1	ND (1.29)	ND (0.985)	ND (0.985)	ND (0.985)	ND (0.985)
ESP Water (Water)	ND (.0033)	ND (.0048)	ND (.0032)	ND (.0018)	ND (.0032)	ND (.0032)	ND (.0032)	ND (.0032)	ND (.0013)	0.0052	ND (.0014)	ND (.0025)	ND (.0013)	ND (.0019)	ND (.0032)
ESP Water (Solids)	15.8	ND (5.66)	6.79	ND (5.66)	ND (5.66)	ND (5.66)	ND (5.66)	ND (5.66)	ND (5.66)	224	ND (28.2)	ND (5.66)	ND (5.66)	ND (5.66)	ND (5.66)
Ash Pit Water (Water)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0003)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)
Ash Pit Water (Solids)			(Sample analysis data not returned from laboratory)												
Effluent Water Field Blank	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0013)	ND (.0010)	ND (.0010)	ND (.0010)	*
Effluent Water Backup Field Blank	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0010)	ND (.0003)	ND (.0010)	ND (.0010)	ND (.0010)	*

[1] Data expressed in ng/g.

* Denotes data not reported by laboratory.

TABLE D-63

AQUEOUS INFLUENT AND EFFLUENT WASTEWATER SAMPLE PRECISION
TCDD ISOMERS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

(Data expressed in ng/g.)

	ISOMERS				
WATER FRACTION	1368	1237/1238	1379	1247/1248/ 1378/1469	1369
ESP Water	0.0038	-------	0.0025	-------	-------
ESP Water Field Duplicate	0.0074	0.0058	0.0057	-------	-------
Precision (RPD)*	64	-------	78	-------	-------
FILTERABLE SOLIDS FRACTION					
ESP Water	1968	1240	945	59.0	-------
ESP Water Field Duplicate	486	1313	-------	-------	65.6
Precision (RPD)	121	6	-------	-------	-------

*Relative percent difference.

IV. SUMMARY OF RESULTS FOR PCDDs AND PCDFs

The analytical data for PCDDs and PCDFs, and for TCDD isomers, are presented in Tables D-64 through D-69, to show the concentrations of these compounds in influent and effluent streams around the Building 703 incinerator on the three sampling days. Those data were combined with the flow rate information appearing in Tables D-64 through D-66, to derive the loadings, in grams per year, of each PCDD and PCDF homologue and TCDD isomer, which are presented in Tables D-70 through D-75. The data for PCDDs and PCDFs were averaged by homologue over the three sampling days and summarized in Figures D-1 through D-10, illustrating the probable destruction, transfer, or formation of each homologue in the incineration process. As described previously, data for loose solid wastes fed to the incinerator could not be gathered as no representative sampling method was available. Figures D-1 through D-10 should be interpreted accordingly.

TABLE D-64
INFLUENT AND EFFLUENT PCDD/PCDF CONCENTRATIONS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

Sample Identification	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	UNITS	FLOW RATE
INFLUENTS														
AIR														
Precombustion Air		58.2/ND*				217/335*		391/628*				21.2/ND*	pg/m^3	30,478 dscfm
LIQUID														
Service Water (Sec. Trtd.)		38.4				198		1260			55.8		ng/L	5.33 x 10^6 L/day
Tittabawassee River Water	(NOT SAMPLED)													
Liquid Waste Nozzle BA													ng/L	9.80 x 10^3 L/day
Liquid Waste Nozzle BB[1]		5790/540	11800/ND	1190/ND	2790/ND	22000/ND		9150/373	845/ND			1240/ND	ng/L	1.92 x 10^4/1.06 x 10^4 L/day
Liquid Waste Nozzle C													ng/L	2.57 x 10^4 L/day
Low-BTU Liquid Waste					10.4								ng/L	4.35 x 10^4 L/day
SOLID														
Loose and Containerized Solid Wastes	(NOT SAMPLED)													
EFFLUENTS														
AIR														
Incinerator Exhaust		47.6	6.49	0.88	0.21	0.93	1.51	86.1	13.6	2.64	0.26	0.06	ng/m^3	30,478 dscfm
LIQUID														
Quench Tower Water								25.0					ng/L	3.86 x 10^6 L/day
Quench Tower Solids		432	54.9	43.7	274	1437	11.0	170	66.4	117	427	379	ng/g	
Venturi/Demister Water								39.3					ng/L	1.47 x 10^6 L/day
Venturi/Demister Solids		238	82.0	55.1	265	1113	8.5	137	100	130	337	284	ng/g	
ESP Water[2]													ng/L	0.95 x 10^6 L/day
ESP Solids													ng/g	
Ash Pit Water													ng/L	0.33 x 10^6 L/day
Ash Pit Solids						323		189					ng/g	
SOLID														
Incinerator Ash		1.17		0.79	6.06	32.7	0.07	9.16	0.07	0.46	1.52	2.57	ng/g	

*Field duplicate sample result.

[1] Two distinct wastes incinerated. Analytical results for both wastes are stated. Loadings (Table D-70) were calculated based on the length of time each waste was burned during the emissions test. ND denotes homologue was not detected.

[2] Sample analysis not returned from laboratory.

TABLE D-65
INFLUENT AND EFFLUENT PCDD/PCDF CONCENTRATIONS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

Sample Identification	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	UNITS	FLOW RATE
INFLUENTS														
AIR														
Precombustion Air		18.0					12.9	12.9	12.5	14.2	108.5	113.7	pg/m^3	31909 dscfm
LIQUID														
Service Water (Sec. Trtd.)		46.4			17.9	187		1420	8.8		16.7	47.7	ng/L	5.30 x 10^6 L/day
Tittabawassee River Water		(NOT SAMPLED)												
Liquid Waste Nozzle BA													ng/L	1.96 x 10^4 L/day
Liquid Waste Nozzle BB[1]		33000/ND	6270/ND	895/ND	3000/ND	11500/ND	284/ND	37000/ND	1780/ND	749/ND	593/ND	625/ND	ng/L	7.42 x 10^3/1.30 x 10^4 L/day
Liquid Waste Nozzle C		60300/21800*	3450/6130*	2610/4240*	3800/5690*	19800/19800*	ND/2100*	36600/18000*	1510/4320*	3510/7130*	8070/8160*	7430/7680*	ng/L	1.27 x 10^4 L/day
Low-BTU Liquid Waste [2]														
SOLID														
Loose and Containerized Solid Wastes		(NOT SAMPLED)												
EFFLUENTS														
AIR														
Incinerator Exhaust		43.8	1.94	0.37	0.84	2.52	1.67	77.0	4.28	1.95	0.55	0.17	ng/m^3	31909 dscfm
LIQUID														
Quench Tower Water								22.3					ng/L	3.89 x 10^6 L/day
Quench Tower Solids		707	99.3	75.3	460	2358	15.4	182	87.5	124	785	641	ng/g	
Venturi/Demister Water								68.2					ng/L	1.41 x 10^6 L/day
Venturi/Demister Solids		307	49.2	27.6	162	707	3.2	168	64.6	82.9	199	283	ng/g	
ESP Water		6.2/18.9*						287/607*					ng/L	0.95 x 10^6 L/day
ESP Solids		4212/1864*	885/393*	147/205*	417/515*	2199/2530*	45.3/47.7*	539/6574*	405/345*	75.7/58.6*	150/161*	200/226*	ng/g	
Ash Pit Water														
Ash Pit Solids		15.9			21.5	94.9		114			10.0	12.5	ng/g	
SOLID														
Incinerator Ash		0.13/0.11*		0.13/0.11*	0.81/0.50	3.2/2.4*	0.02/ND*	0.59/0.26*		0.04/0.04*	0.45/0.25*	0.57/0.40*	ng/g	

*Field duplicate sample result.

[1] Two distinct wastes incinerated. Analytical results for both wastes are stated. Loadings (Table D-71) were calculated based on the length of time each waste was burned during the emissions test. ND denotes homologue was not detected.

[2] No waste incinerated.

TABLE D-66
INFLUENT AND EFFLUENT PCDD/PCDF CONCENTRATIONS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

Sample Identification	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	UNITS	FLOW RATE
INFLUENTS														
AIR														
Precombustion Air		38.9			98.1	306.5		206.6			37.4	30.9	pg/m^3	33.599 dscfm
LIQUID														
Service Water (Sec. Trtd.)														5.10 x 10^6 L/day
Tittabawassee River Water		(NOT SAMPLED)												
Liquid Waste Nozzle BA		5880	808			1210	237	6480	178				ng/L	1.88 x 10^4 L/day
Liquid Waste Nozzle BB		835						181					ng/L	3.27 x 10^4 L/day
Liquid Waste Nozzle C		29.3/22.8*			181/132*	753/570*		33.9/46.4*					ng/L	1.91 x 10^4 L/day
Low-BTU Liquid Waste													ng/L	5.17 x 10^4 L/day
SOLID														
Loose and Containerized Solid Wastes		(NOT SAMPLED)												
EFFLUENTS														
AIR														
Incinerator Exhaust		199				0.47		288	0.17	9.68			ng/m^3	33,599 dscfm
LIQUID														
Quench Tower Water								5.80					ng/L	3.91 x 10^6 L/day
Quench Tower Solids		73.9			69.0	236		830	7.09	16.1	125	103	ng/g	
Venturi/Demister Water								15.7					ng/L	1.19 x 10^6 L/day
Venturi/Demister Solids		53.6	17.5	7.35	44.3	261	2.05	723	22.3	19.7	69.1	84.8	ng/g	
ESP Water		5.2						99.5					ng/L	0.95 x 10^6 L/day
ESP Solids		247	61.5	20.3	96.0	423	9.70	90.0	47.0	14.7	68.2	82.1	ng/g	
Ash Pit Water													ng/L	0.33 x 10^6 L/day
Ash Pit Solids [1]													ng/g	
SOLID														
Incinerator Ash		0.07			0.08	0.27		0.54				0.08	ng/g	

*Field duplicate sample result.

[1] Sample analysis not returned from laboratory.

TABLE D-67
INFLUENT AND EFFLUENT TCDD ISOMER CONCENTRATIONS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

Sample Identification	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289	UNITS
INFLUENTS																
AIR																
Precombustion Air	44.2	14.0														pg/m^3
LIQUID																
Service Water (Sec. Trtd.)	18.0	9.9								10.5						ng/L
Tittabawassee River Water		(NOT SAMPLED)														ng/L
Liquid Waste Nozzle BA																ng/L
*Liquid Waste Nozzle BB	1189/276	4108/272								493/ND						ng/L
Liquid Waste Nozzle C																ng/L
Low-BTU Liquid Waste																ng/L
SOLID																
Loose and Containerized Solid Wastes		(NOT SAMPLED)														
EFFLUENTS																
AIR																
Incinerator Exhaust	20.4	13.0		1.69						12.6						ng/m^3
LIQUID																
Quench Tower Water																ng/L
Quench Tower Solids	183	113		7.32						128						ng/g
Venturi/Demister Water																ng/L
Venturi/Demister Solids	113	7.83								117						ng/g
ESP Water																ng/L
ESP Solids																ng/g
Ash Pit Water																ng/L
Ash Pit Solids																ng/g
SOLID																
Incinerator Ash	0.62	0.25		0.04						0.27						ng/g

*Two distinct wastes incinerated. Analytical results for both wastes are stated in a manner similar to that in Table D-64.

TABLE D-68
INFLUENT AND EFFLUENT TCDD ISOMER CONCENTRATIONS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

Sample Identification	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289	UNITS
INFLUENTS																
AIR																
Precombustion Air		4.34		1.63				0.98	0.82	5.05	5.18					pg/m^3
LIQUID																
Service Water (Sec. Trtd.)	20.6	16.1								9.70						ng/L
Tittabawassee River Water		(NOT SAMPLED)														ng/L
Liquid Waste Nozzle BA																ng/L
Liquid Waste Nozzle BB [1]	21800/ND	10300/ND		440/ND						440/ND						ng/L
Liquid Waste Nozzle C	39900/8840*	20400/10800*		ND/430*						ND/1400*		ND/320*				ng/L
Low-BTU Liquid Waste [2]																ng/L
SOLID																
Loose and Containerized Solid Wastes		(NOT SAMPLED)														
EFFLUENTS																
AIR																
Incinerator Exhaust	17.2	13.0		0.21						13.3						ng/m^3
LIQUID																
Quench Tower Water																ng/L
Quench Tower Solids	290	183		14.5						220						ng/g
Venturi/Demister Water																ng/L
Venturi/Demister Solids	130	85.5		7.77						84.2						ng/g
ESP Water	3.8/7.4	2.5/5.7								ND/5.8						ng/L
ESP Solids	1968/486*	945/ND*	ND/65.6*	59.0/ND*						1240/1313*						ng/g
Ash Pit Water																ng/L
Ash Pit Solids	7.47	3.97		0.94						3.50						ng/g
SOLID																
Incinerator Ash	0.06/0.06*	0.04/0.03*		0.01/ND*						0.02/0.02*						ng/g

*Field duplicate sample.

[1] Two distinct wastes incinerated. Analytical results for both wastes are stated, in similar manner as in Table D-65.

[2] No waste incinerated.

TABLE D-69
INFLUENT AND EFFLUENT TCDD ISOMER CONCENTRATIONS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

Sample Identification	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289	UNITS
INFLUENTS																
AIR																
Precombustion Air	24.0	7.58		2.45					0.98	3.92						pg/m³
LIQUID																
Service Water (Sec. Trtd.)																ng/L
Tittabawassee River Water		(NOT SAMPLED)														ng/L
Liquid Waste Nozzle BA																ng/L
Liquid Waste Nozzle BB	4050	1840														ng/L
Liquid Waste Nozzle C	636	199														ng/L
Low-BTU Liquid Waste	19.1/14.4*	6.23/6.00*								4.00/2.40*						ng/L
SOLID																
Loose and Containerized Solid Wastes		(NOT SAMPLED)														
EFFLUENTS																
AIR																
Incinerator Exhaust	129	70.7		0.06						0.46						ng/m³
LIQUID																
Quench Tower Water																ng/L
Quench Tower Solids	26.9	24.7								22.2						ng/g
Venturi/Demister Water																ng/L
Venturi/Demister Solids	28.2		2.01							26.1						ng/g
ESP Water										5.2						ng/L
ESP Solids	15.8		6.79							224						ng/g
Ash Pit Water																ng/L
Ash Pit Solids [1]																ng/g
SOLID																
Incinerator Ash	0.04	0.02								0.01						ng/g

*Field duplicate sample results.

[1] Sample analysis not returned from laboratory.

TABLE D-70

INFLUENT AND EFFLUENT PCDD/PCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84
(in grams per year)

Sample Identification	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	UNITS
INFLUENTS													
AIR													
Precombustion Air		0.026				0.099		0.18				0.003	
LIQUID													
Service Water (Sec. Trtd.)		74.8				385		2451			108		
Tittabawassee River Water		(NOT SAMPLED)											
Liquid Waste Nozzle BA													
Liquid Waste Nozzle BB[1]		42.7	82.7	8.32	195	154		65.6	5.92			8.69	
Liquid Waste Nozzle C													
Low-BTU Liquid Waste					0.16								
SOLID													
Loose and Containerized Solid Wastes		(NOT SAMPLED)											
TOTAL INFLUENTS (grams/year)		117.5	82.7	8.32	195	539		2517	5.92		108	8.69	
EFFLUENTS													
AIR													
Incinerator Exhaust		21.6	2.94	0.40	0.09	0.44	0.69	38.9	6.14	1.20	0.12	0.03	
LIQUID													
Quench Tower Water								35.2					
Quench Tower Solids		4.32	5.50	4.37	27.4	144	1.10	17.0	6.65	11.7	42.7	37.9	
Venturi/Demister Water								21.1					
Venturi/Demister Solids		9.83	3.39	2.26	10.9	46.0	0.35	5.66	4.14	5.37	13.9	11.7	
ESP Water[2]													
ESP Solids[2]													
Ash Pit Water													
Ash Pit Solids						0.12		0.07					
SOLID													
Incinerator Ash		3.84-5.12		2.60-3.47	19.9-26.5	107-143	0.22-0.29	30.0-40.1	0.22-0.30	1.49-1.99	4.99-6.65	8.43-11.2	
TOTAL EFFLUENTS (grams year)		40.2	11.8	10.1	61.6	316	2.41	153	17.2	20.0	62.5	59.4	

[1] Total of two wastes incinerated.

[2] Sample analysis not returned from laboratory.

TABLE D-71
INFLUENT AND EFFLUENT PCDD/PCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84
(in grams per year)

Sample Identification	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	UNITS
INFLUENTS													
AIR													
Precombustion Air	0.002	0.008					0.006	0.006	0.006	0.007	0.005	0.005	
LIQUID													
Service Water (Sec. Trtd.)		89.8			34.7	362		2748	17.2		32.5	92.3	
Tittabawassee River Water		(NOT SAMPLED)											
Liquid Waste Nozzle BA													
Liquid Waste Nozzle BB [1]		89.4	17.0	2.42	8.14	31.1	0.77	100	4.82	2.03	1.61	1.69	
Liquid Waste Nozzle C		280	16.0	12.1	17.6	91.8		171	7.01	16.3	37.4	34.5	
Low-BTU Liquid Waste [2]													
SOLID													
Loose and Containerized Solid Wastes		(NOT SAMPLED)											
TOTAL INFLUENTS (grams/year)		459	33	14.5	60.4	485	0.78	3019	29.0	18.3	71.5	128	
EFFLUENTS													
AIR													
Incinerator Exhaust		20.8	0.92	0.17	0.40	1.20	0.79	36.6	2.03	0.93	0.26	0.08	
LIQUID													
Quench Tower Water								31.7					
Quench Tower Solids		111	15.6	11.8	72.5	371	2.41	28.7	13.8	19.6	124	101	
Venturi/Demister Water								35.1					
Venturi/Demister Solids		20.9	3.35	1.87	10.98	48.0	0.22	11.4	4.42	5.63	13.5	19.2	
ESP Water		2.15						99.5					
ESP Solids		23.4	4.89	0.82	2.31	12.2	0.25	2.99	2.25	0.42	0.83	1.11	
Ash Pit Water													
Ash Pit Solids		0.20			0.31	1.46		1.78			0.14	0.20	
SOLID													
Incinerator Ash		0.43-0.57		0.42-0.56	2.65-3.53	10.4-13.9	0.05-0.07	1.95-2.60		0.15-0.19	1.47-1.97	1.88-2.51	
TOTAL EFFLUENTS (grams/year)		179	24.8	15.2	89.6	446	3.73	250	22.5	26.8	140	124	

*Field duplicate sample result.

[1] Total of two wastes incinerated.

[2] No waste incinerated.

TABLE D-72
INFLUENT AND EFFLUENT PCDD/PCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84
(in grams per year)

Sample Identification	2378-TCDD	Total TCDD	Total PeCDD	Total HxCDD	Total HpCDD	OCDD	2378-TCDF	Total TCDF	Total PeCDF	Total HxCDF	Total HpCDF	OCDF	UNITS
INFLUENTS													
AIR													
Precombustion Air		0.019			0.047	0.153		0.102			0.019	0.015	
LIQUID													
Service Water (Sec. Trtd.)													
Tittabawassee River Water		(NOT SAMPLED)											
Liquid Waste Nozzle BA													
Liquid Waste Nozzle BB		70.2	9.64			14.5	2.83	77.3	2.12				
Liquid Waste Nozzle C		5.82						1.26					
Low-BTU Liquid Waste		0.55			3.40	14.1		0.62					
SOLID													
Loose and Containerized Solid Wastes		(NOT SAMPLED)											
TOTAL INFLUENTS (grams/year)		76.6	9.64		3.40	28.8	2.83	79.3	2.12		0.019	0.015	
EFFLUENTS													
AIR													
Incinerator Exhaust		2.46				0.23		47.2	0.09				
LIQUID													
Quench Tower Water								8.28					
Quench Tower Solids		13.4			11.1	42.8		151	1.27	2.94	22.6	18.7	
Venturi/Demister Water								6.82					
Venturi/Demister Solids		4.10	1.29	0.54	3.26	19.1	0.14	53.0	1.61	1.46	5.05	6.24	
ESP Water		1.80						34.5					
ESP Solids		20.7	5.22	1.69	7.98	31.7	0.78	7.48	3.95	1.27	5.65	6.78	
Ash Pit Water [1]													
Ash Pit Solids [1]													
SOLID													
Incinerator Ash		0.23-0.31			0.25-0.33	0.87-1.16		1.77-2.36				0.26-0.34	
TOTAL EFFLUENTS (grams/year)		42.7	6.51	2.23	22.6	94.6	0.92	310	6.92	5.7	33.3	32.0	

[1] Sample analysis not returned from laboratory.

TABLE D-73
INFLUENT AND EFFLUENT TCDD ISOMER LOADINGS (in grams per year)
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/28/84

Sample Identification	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
INFLUENTS															
AIR															
Precombustion Air	0.02	0.01													
LIQUID															
Service Water (Sec. Trtd.)	35.1	19.3								20.5					
Tittabawassee River Water			(NOT SAMPLED)												
Liquid Waste Nozzle BA															
[1] Liquid Waste Nozzle BB	9.39	29.9								3.45					
Liquid Waste Nozzle C															
Low-BTU Liquid Waste															
SOLID															
Loose and Containerized Solid Wastes			(NOT SAMPLED)												
TOTAL INFLUENTS (grams/year)	44.5	49.2								24.0					
EFFLUENTS															
AIR															
Incinerator Exhaust	9.18	5.85		0.80						5.68					
LIQUID															
Quench Tower Water															
Quench Tower Solids	18.2	11.3		0.74						12.80					
Venturi/Demister Water															
Venturi/Demister Solids	4.68		0.32							4.84					
ESP Water															
ESP Solids															
Ash Pit Water															
Ash Pit Solids															
SOLID															
Incinerator Ash	2.03-2.71	0.81-1.09		0.12-0.16						0.87-1.17					
TOTAL EFFLUENTS (grams/year)	34.5	18.1	0.32	1.68						24.3					

[1] Total of two wastes incinerated.

TABLE D-74
INFLUENT AND EFFLUENT TCDD ISOMER LOADINGS (in grams per year)
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
8/30/84

Sample Identification	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
INFLUENTS															
AIR															
Precombustion Air		0.002		0.0008		0.0005			0.0004	0.002	0.002				
LIQUID															
Service Water (Sec. Trtd.)	45.0	35.0								9.78					
Tittabawassee River Water		(NOT SAMPLED)													
Liquid Waste Nozzle BA															
[1] Liquid Waste Nozzle BB	59.1	27.9		1.18						1.18					
Liquid Waste Nozzle C															
Low-BTU Liquid Waste [2]	185	94.6													
SOLID															
Loose and Containerized Solid Wastes		(NOT SAMPLED)													
TOTAL INFLUENTS (grams/year)	289	158		1.18		0.0005			0.0004	11.0	0.002				
EFFLUENTS															
AIR															
Incinerator Exhaust	7.98	6.02		0.10						6.16					
LIQUID															
Quench Tower Water															
Quench Tower Solids	45.7	28.9		2.28						34.6					
Venturi/Demister Water															
Venturi/Demister Solids	8.83	5.81		0.53						5.72					
ESP Water	1.32	0.87													
ESP Solids	10.9	5.27		0.33						6.87					
Ash Pit Water															
Ash Pit Solids	0.11	0.06		0.01						0.06					
SOLID															
Incinerator Ash	0.21-0.28	0.11-0.15		0.03-0.03						0.08-0.10					
TOTAL EFFLUENTS (grams/year)	75.1	47.1		3.30						53.5					

[1] Total of two wastes incinerated. [2] No waste incinerated.

TABLE D-75
INFLUENT AND EFFLUENT TCDD ISOMER LOADINGS (in grams per year)
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR
9/5/84

Sample Identification	1368	1379	1369	1247 1248 1378 1469	1246 1249	1268 1278	1478	1268 1279	1234 1236 1269	1237 1238	2378	1239	1278 1279	1267	1289
INFLUENTS															
AIR															
Precombustion Air	0.012	0.004	0.001						0.0005	0.002					
LIQUID															
Service Water (Sec. Trtd.)															
Tittabawassee River Water		(NOT SAMPLED)													
Liquid Waste Nozzle BA															
Liquid Waste Nozzle BB	48.3	22.0													
Liquid Waste Nozzle C	4.43	1.39													
Low-BTU Liquid Waste	0.36	0.12								0.07					
SOLID															
Loose and Containerized Solid Wastes		(NOT SAMPLED)													
TOTAL INFLUENTS (grams/year)	53.1	23.5	0.001						0.0005	0.073					
EFFLUENTS															
AIR															
Incinerator Exhaust	64.3	35.3		0.03						0.21					
LIQUID															
Quench Tower Water															
Quench Tower Solids	4.90	4.46								4.03					
Venturi/Demister Water															
Venturi/Demister Solids	2.07		0.14							1.92					
ESP Water										1.80					
ESP Solids	1.34		0.56							18.6					
Ash Pit Water [1]															
Ash Pit Solids [1]															
SOLID															
Incinerator Ash	0.11-0.15	0.08-0.10								0.05-0.06					
TOTAL EFFLUENTS (grams/year)	72.7	39.9	0.70	0.03						26.6					

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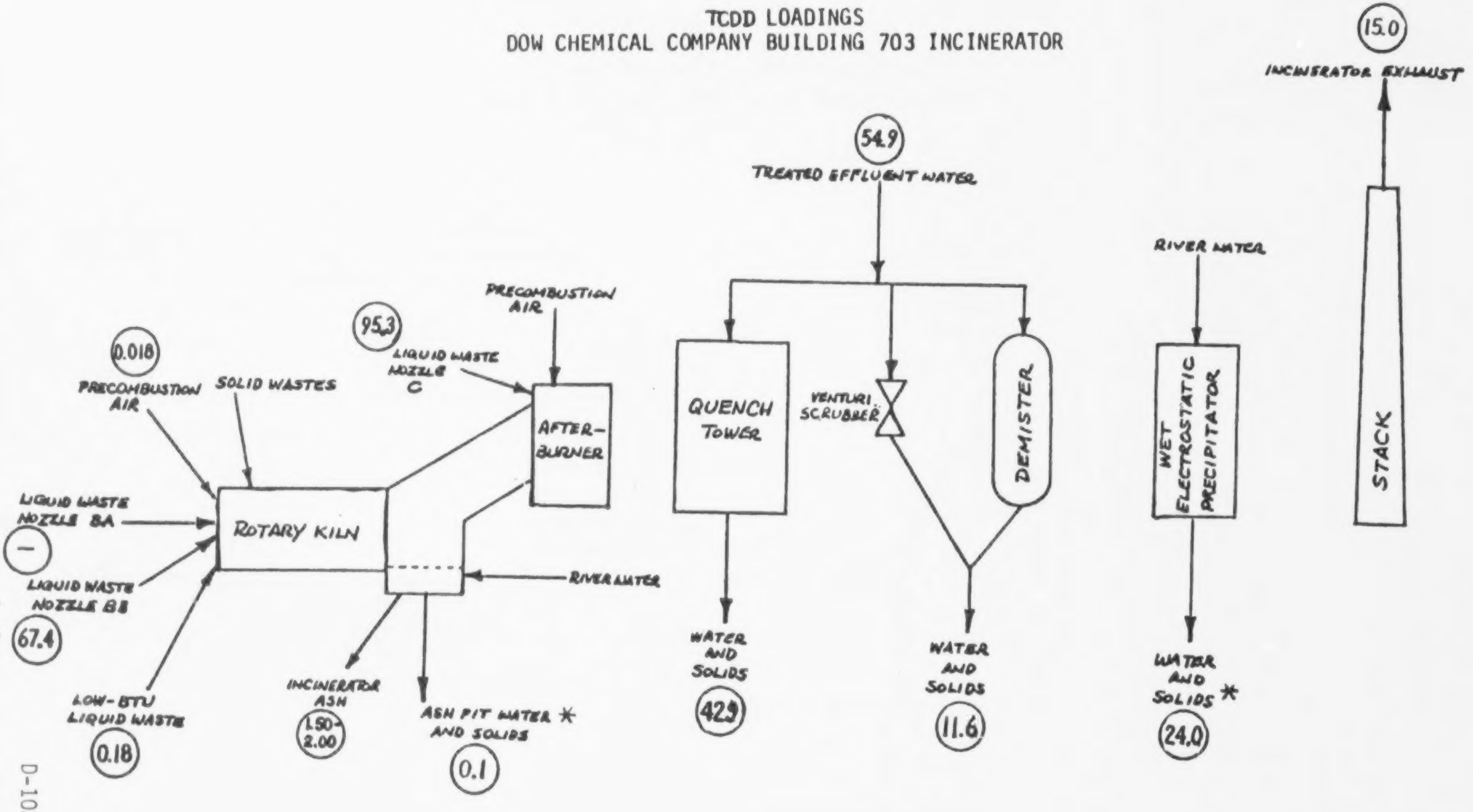
[1] Sample analysis not returned from laboratory.

FIGURE D-1

TCDD LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


15.0
INCINERATOR EXHAUST
54.9
TREATED EFFLUENT WATER
RIVER WATER
PRECOMBUSTION AIR
95.3
LIQUID WASTE NOZZLE C
0.018
PRECOMBUSTION AIR
SOLID WASTES
AFTER-BURNER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WET ELECTROSTATIC PRECIPITATOR
STACK
LIQUID WASTE NOZZLE 8A
ROTARY KILN
—
LIQUID WASTE NOZZLE 8B
RIVER WATER
67.4
WATER AND SOLIDS
WATER AND SOLIDS
WATER AND SOLIDS *
INCINERATOR ASH
LOW-BTU LIQUID WASTE
ASH PIT WATER AND SOLIDS *
42.9
11.6
24.0
1.50-2.00
0.18
0.1

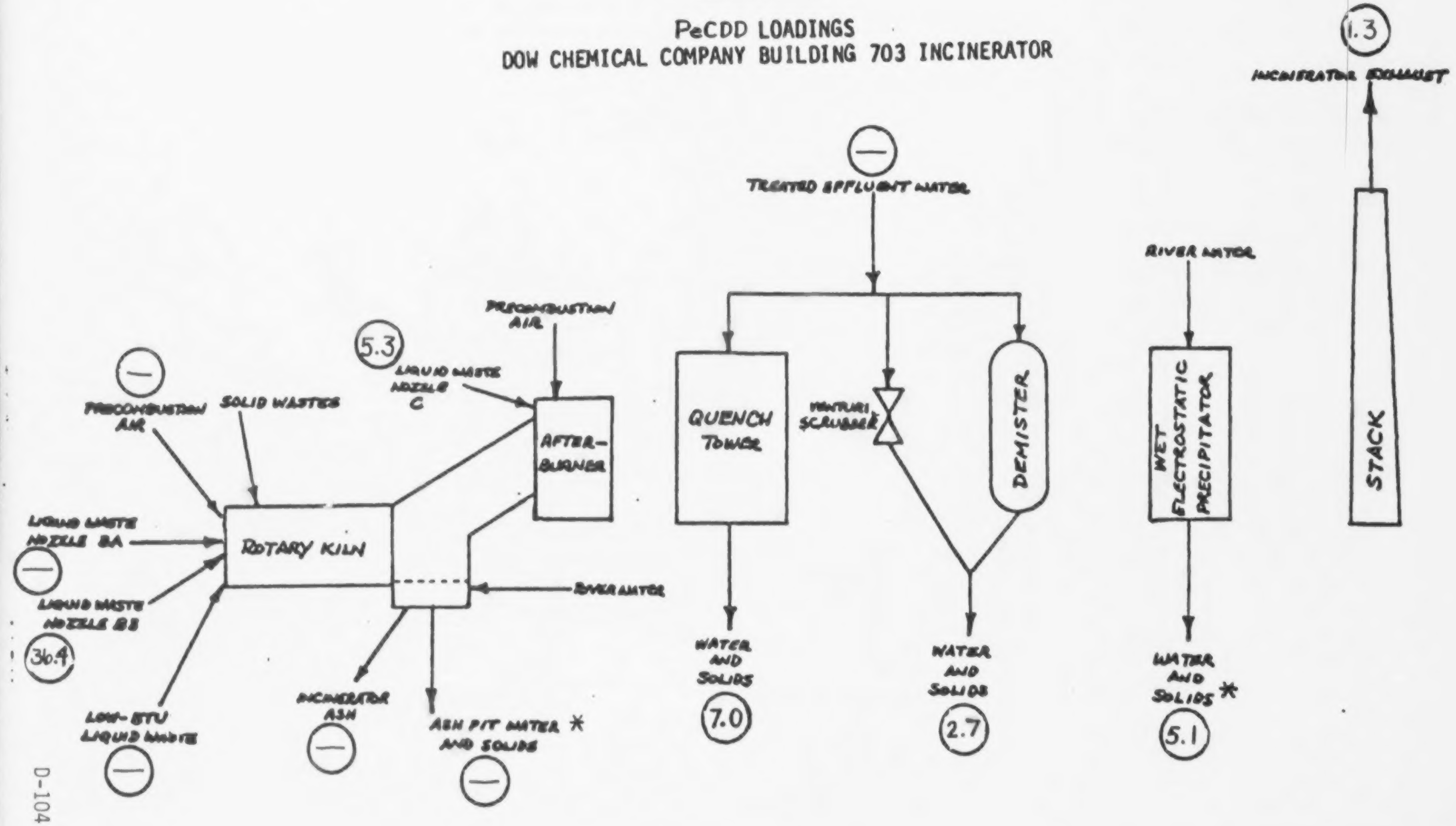
NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF TCDD		
In		Out
218	(gm/yr)	87.3

FIGURE D-2

PeCDD LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


1.3
INCINERATOR EXHAUST
STACK
—
TREATED EFFLUENT WATER
RIVER WATER
WET ELECTROSTATIC PRECIPITATOR
WATER AND SOLIDS *
5.1
QUENCH TOWER
WATER AND SOLIDS
7.0
VENTURI SCRUBBER
DEMISTER
WATER AND SOLIDS
2.7
PRECOMBUSTION AIR
5.3
LIQUID WASTE NOZZLE C
AFTER-BURNER
—
PRECOMBUSTION AIR
SOLID WASTES
ROTARY KILN
LIQUID WASTE NOZZLE 8A
—
LIQUID WASTE NOZZLE 8B
36.4
LOW-BTU LIQUID WASTE
—
RIVER WATER
INCINERATOR ASH
—
ASH PIT WATER AND SOLIDS *
—

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

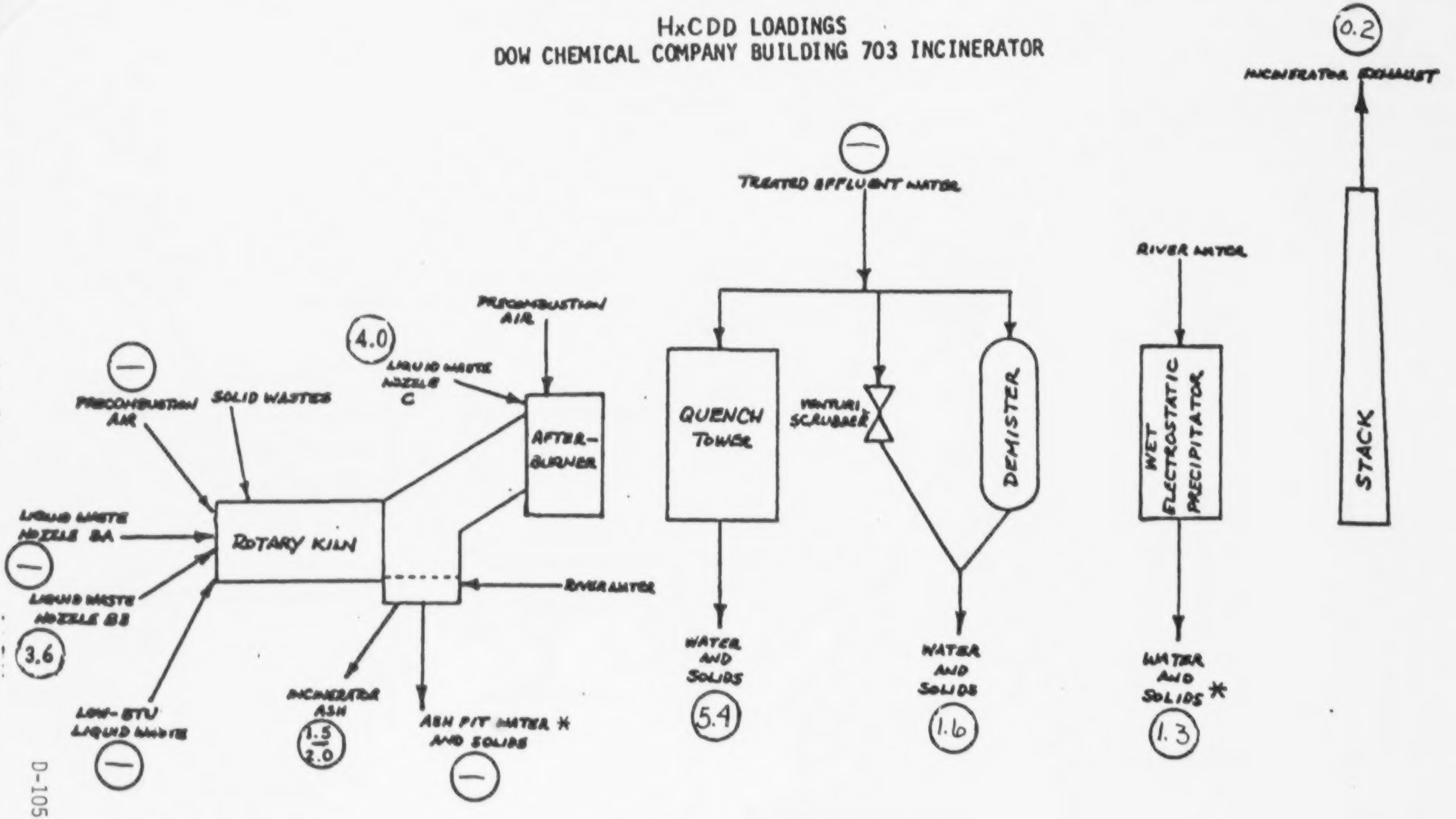
* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF PeCDD

In		Out
42	(g/yr)	14

FIGURE D-3

HxCDD LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


0.2
INCINERATOR EXHAUST
STACK
TREATED EFFLUENT WATER
—
RIVER WATER
WET ELECTROSTATIC PRECIPITATOR
WATER AND SOLIDS *
1.3
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WATER AND SOLIDS
5.4
WATER AND SOLIDS
1.6
PRECOMBUSTION AIR
4.0
LIQUID WASTE NOZZLE C
AFTER-BURNER
—
PRECOMBUSTION AIR
SOLID WASTES
LIQUID WASTE NOZZLE 8A
—
ROTARY KILN
LIQUID WASTE NOZZLE 8B
3.6
LOW-BTU LIQUID WASTE
—
RIVER WATER
INCINERATOR ASH
1.5
2.0
ASH PIT WATER * AND SOLIDS
—

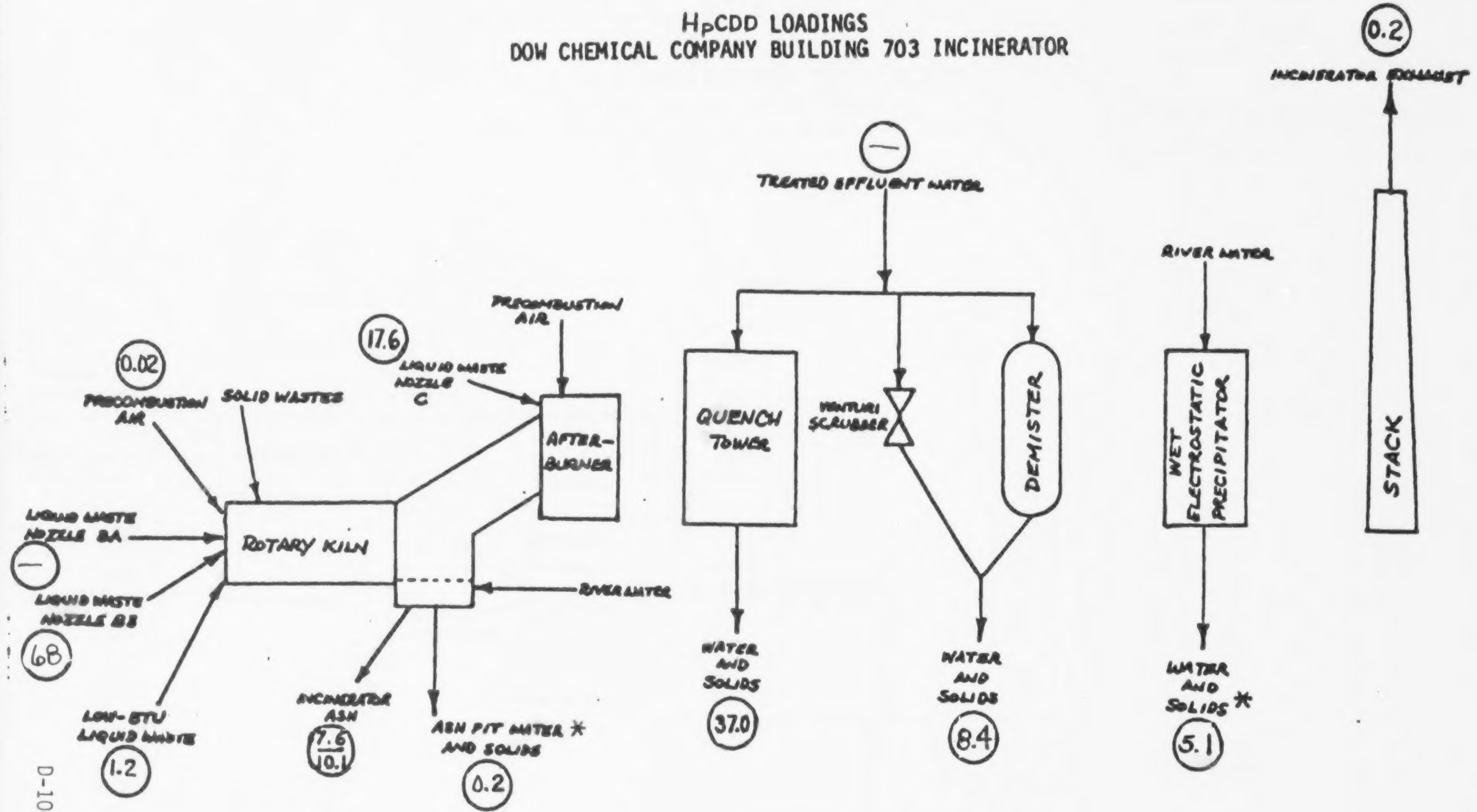
NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF HxCDD		
In		Out
7.6	(gm/yr)	9.2

FIGURE D-4

H_pCDD LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


0.2
INCINERATOR EXHAUST
TREATED EFFLUENT WATER
—
RIVER WATER
PRECOMBUSTION AIR
17.6
LIQUID WASTE NOZZLE C
0.02
PRECOMBUSTION AIR
SOLID WASTES
AFTER-BURNER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WET ELECTROSTATIC PRECIPITATOR
STACK
LIQUID WASTE NOZZLE 8A
—
ROTARY KILN
RIVER WATER
LIQUID WASTE NOZZLE 8B
68
LOW-BTU LIQUID WASTE
1.2
INCINERATOR ASH
7.6/10.1
ASH PIT WATER AND SOLIDS *
0.2
WATER AND SOLIDS
37.0
WATER AND SOLIDS
8.4
WATER AND SOLIDS *
5.1

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

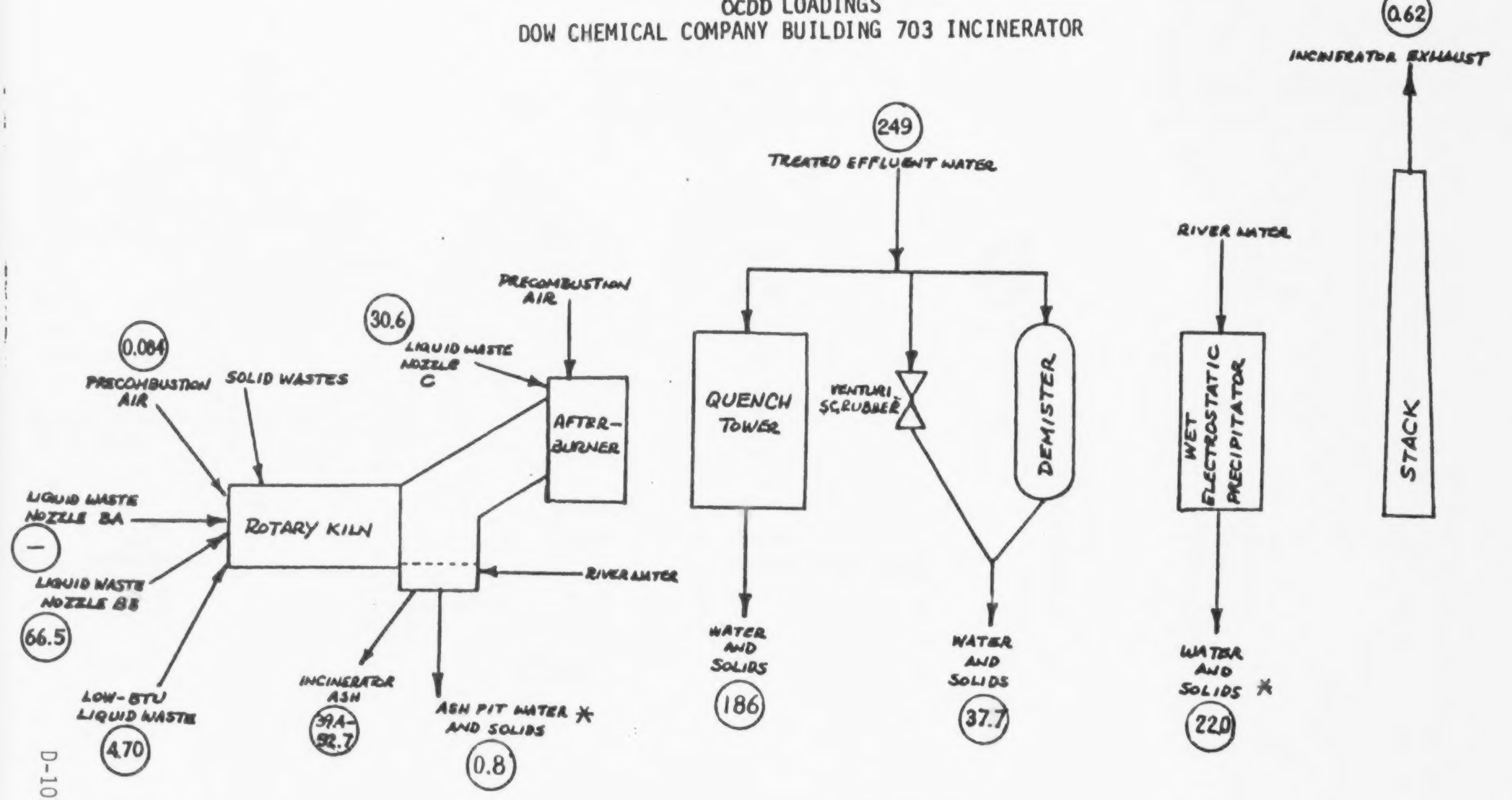
* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF H_pCDD

In		Out
86	(gm/yr)	58

FIGURE D-5

OCDD LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


0.62
INCINERATOR EXHAUST
249
TREATED EFFLUENT WATER
RIVER WATER
PRECOMBUSTION AIR
30.6
LIQUID WASTE NOZZLE C
0.084
PRECOMBUSTION AIR
SOLID WASTES
AFTER-BURNER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WET ELECTROSTATIC PRECIPITATOR
STACK
LIQUID WASTE NOZZLE BA
—
ROTARY KILN
RIVER WATER
LIQUID WASTE NOZZLE BB
66.5
LOW-BTU LIQUID WASTE
4.70
INCINERATOR ASH
39.4-52.7
ASH PIT WATER AND SOLIDS *
0.8
WATER AND SOLIDS
186
WATER AND SOLIDS
37.7
WATER AND SOLIDS *
22.0

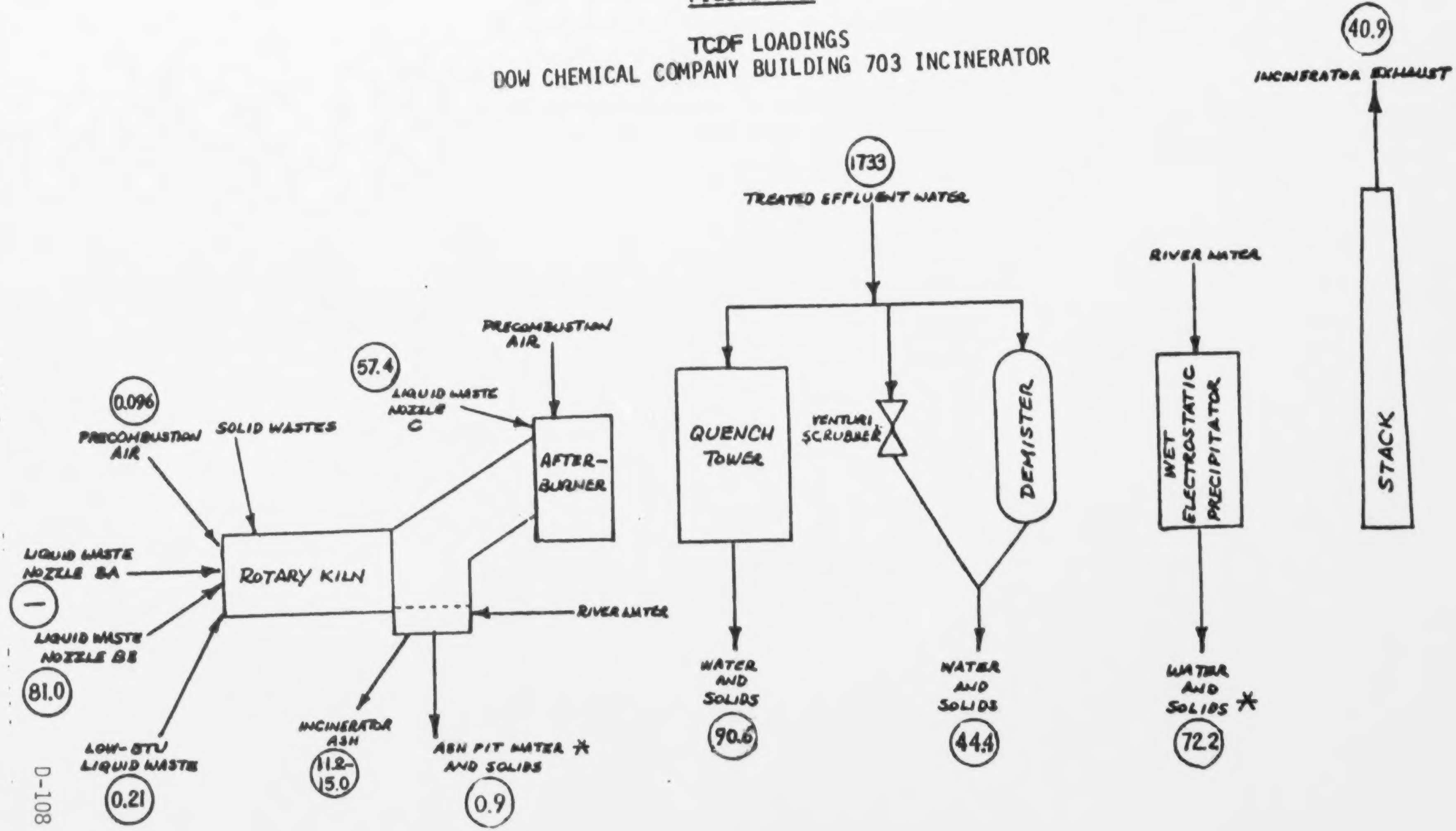
NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF OCDD		
In		Out
351	(gm/yr)	286

FIGURE D-6

TCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


40.9
INCINERATOR EXHAUST
STACK
1733
TREATED EFFLUENT WATER
RIVER WATER
WET ELECTROSTATIC PRECIPITATOR
WATER AND SOLIDS *
72.2
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WATER AND SOLIDS
90.6
WATER AND SOLIDS
44.4
PRECOMBUSTION AIR
57.4
LIQUID WASTE NOZZLE C
AFTER-BURNER
0.096
PRECOMBUSTION AIR
SOLID WASTES
ROTARY KILN
RIVER WATER
LIQUID WASTE NOZZLE 8A
—
LIQUID WASTE NOZZLE 8B
81.0
LOW-BTU LIQUID WASTE
0.21
INCINERATOR ASH
11.2-15.0
ASH PIT WATER * AND SOLIDS
0.9

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

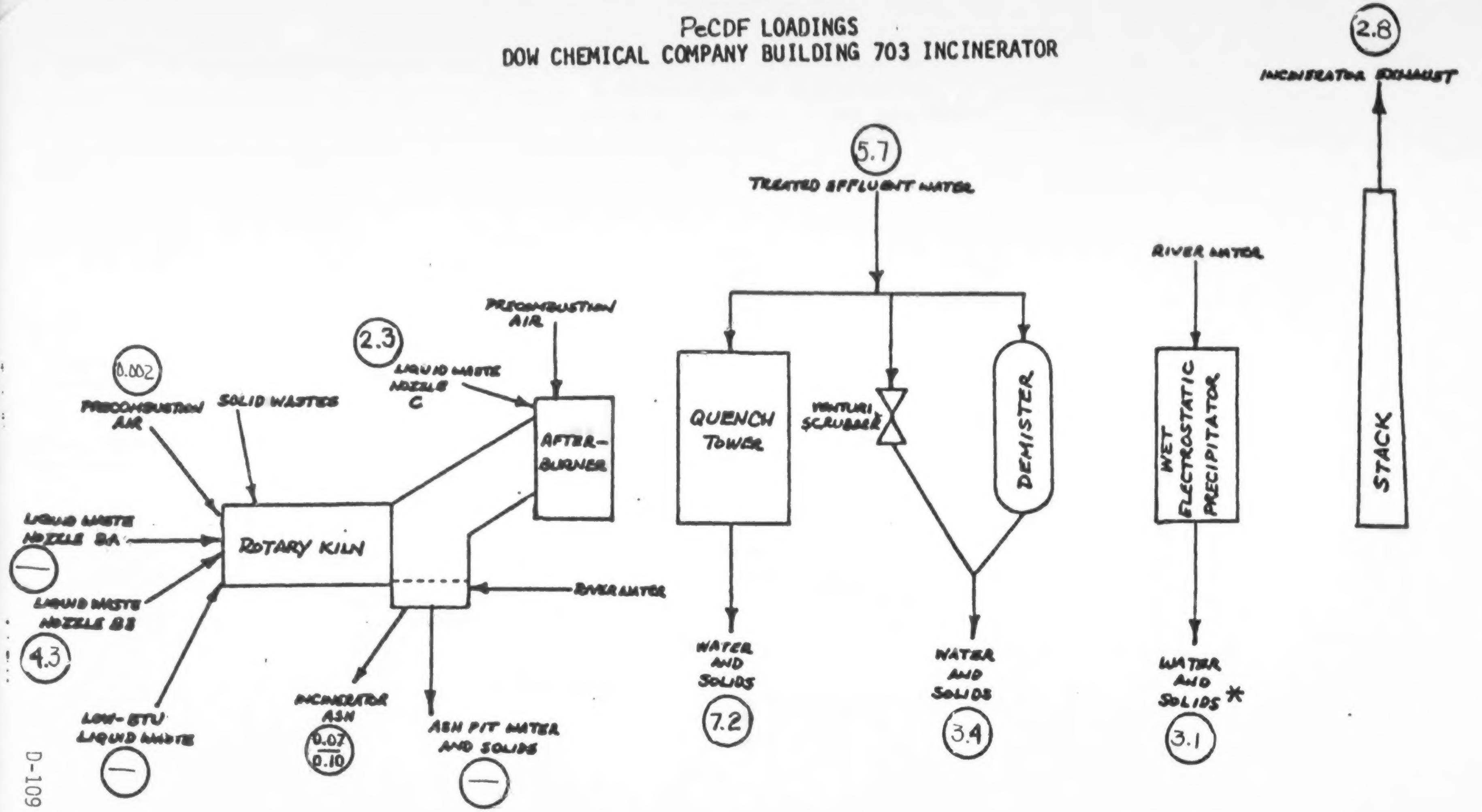
* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF TCDF

In		Out
1872	(gm/yr)	238

FIGURE D-7

PeCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR



0.002
PRECOMBUSTION AIR
SOLID WASTES
LIQUID WASTE NOZZLE 8A
—
LIQUID WASTE NOZZLE 8B
4.3
LOW-BTU LIQUID WASTE
—
ROTARY KILN
2.3
LIQUID WASTE NOZZLE C
PRECOMBUSTION AIR
AFTER-BURNER
RIVER WATER
INCINERATOR ASH
0.07
0.10
ASH PIT WATER AND SOLIDS
—
5.7
TREATED EFFLUENT WATER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WATER AND SOLIDS
7.2
WATER AND SOLIDS
3.4
RIVER WATER
WET ELECTROSTATIC PRECIPITATOR
WATER AND SOLIDS *
3.1
2.8
INCINERATOR EXHAUST
STACK

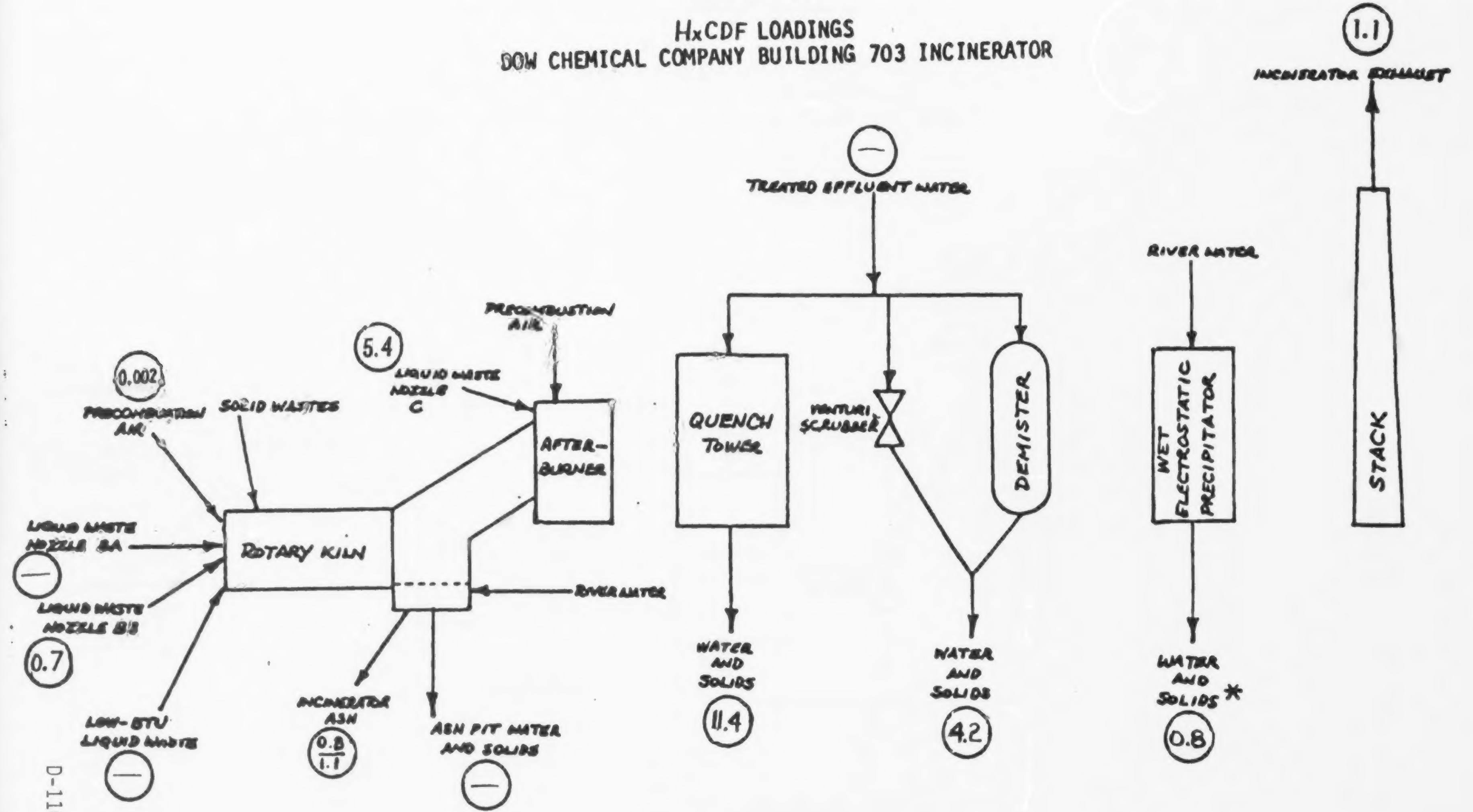
NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF PeCDF		
In		Out
12.3	(g/yr)	15.5

FIGURE D-8

HxCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR



1.1
INCINERATOR EXHAUST
—
TREATED EFFLUENT WATER
RIVER WATER
PRECOMBUSTION AIR
5.4
LIQUID WASTE NOZZLE C
0.002
PRECOMBUSTION AIR
SOLID WASTES
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WET ELECTROSTATIC PRECIPITATOR
STACK
AFTER-BURNER
LIQUID WASTE NOZZLE 8A
ROTARY KILN
—
RIVER WATER
LIQUID WASTE NOZZLE 8B
0.7
WATER AND SOLIDS
WATER AND SOLIDS
WATER AND SOLIDS *
INCINERATOR ASH
LOW-BTU LIQUID WASTE
ASH PIT WATER AND SOLIDS
11.4
4.2
0.8
0.8/1.1
—
—

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

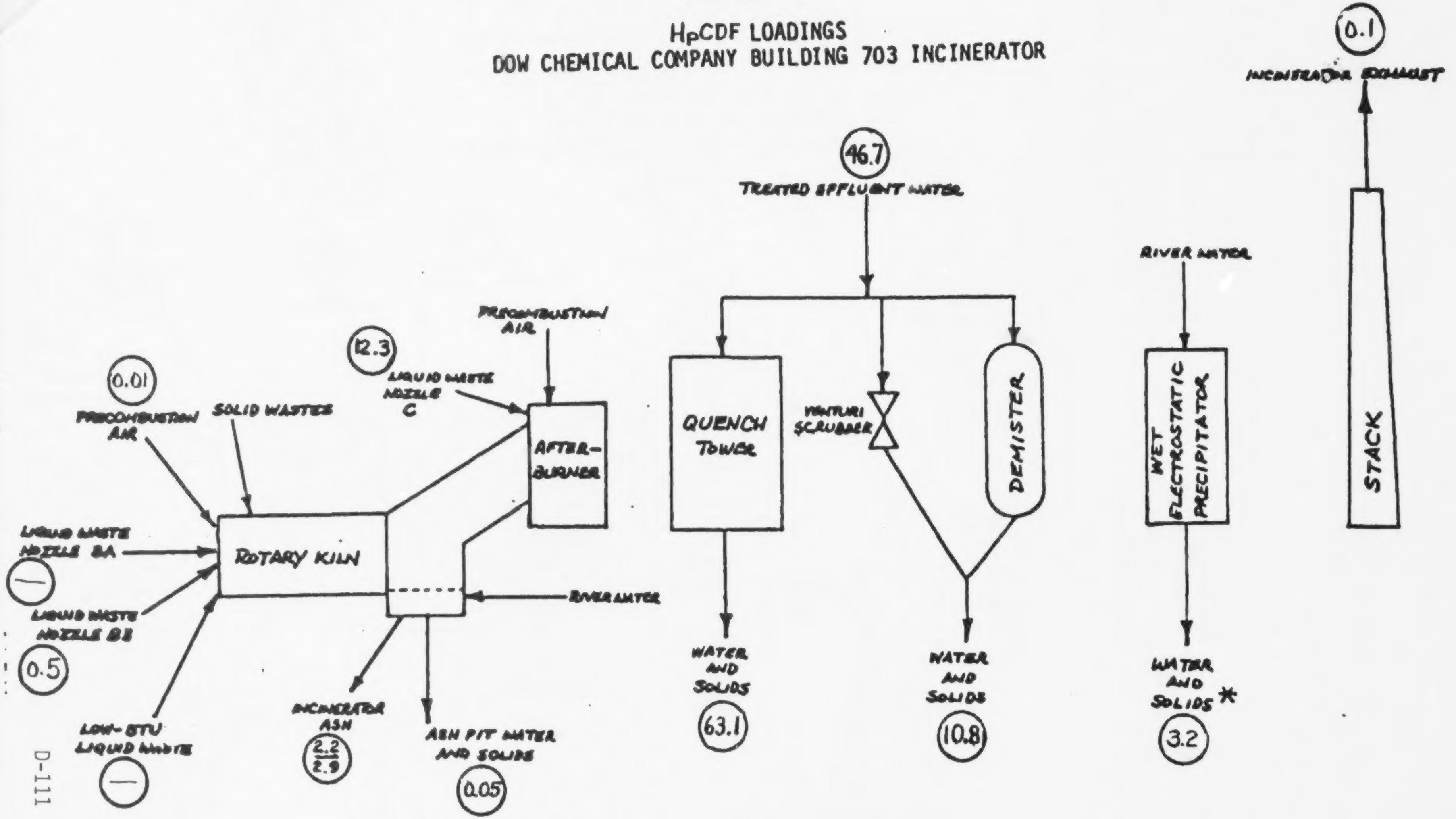
* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF HxCDF

In		Out
6.1	(gm/yr)	17.5

FIGURE D-9

H_pCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


0.1
INCINERATOR EXHAUST
STACK
46.7
TREATED EFFLUENT WATER
RIVER WATER
PRECOMBUSTION AIR
12.3
LIQUID WASTE NOZZLE C
0.01
PRECOMBUSTION AIR
SOLID WASTES
AFTER-BURNER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WET ELECTROSTATIC PRECIPITATOR
LIQUID WASTE NOZZLE 8A
ROTARY KILN
RIVER WATER
LIQUID WASTE NOZZLE 8B
0.5
LOW-BTU LIQUID WASTE
INCINERATOR ASH
2.2
2.9
ASH PIT WATER AND SOLIDS
0.05
WATER AND SOLIDS
63.1
WATER AND SOLIDS
10.8
WATER AND SOLIDS *
3.2

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

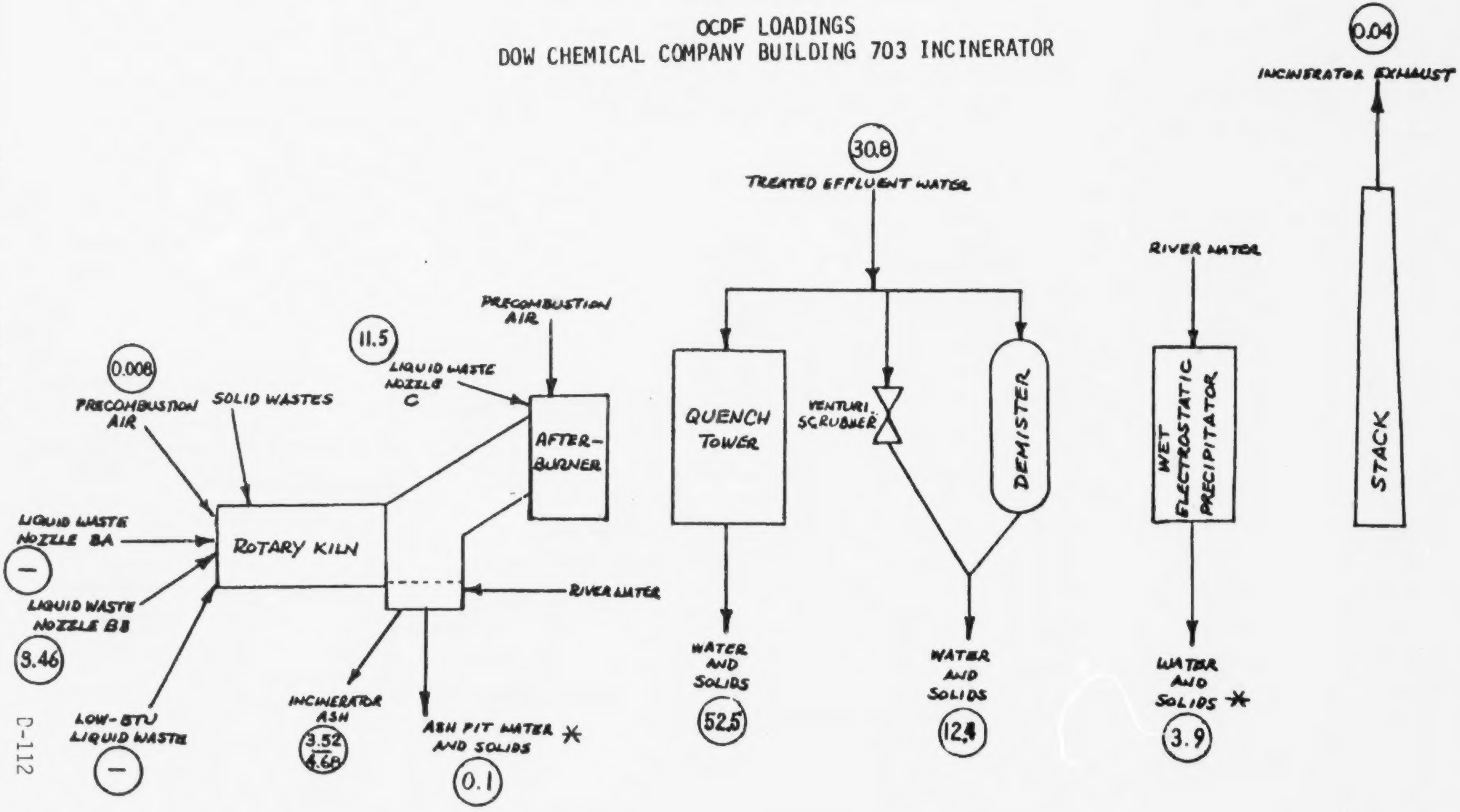
* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF H_pCDF

In		Out
60	(g/yr)	79

FIGURE D-10

OCDF LOADINGS
DOW CHEMICAL COMPANY BUILDING 703 INCINERATOR


0.04
INCINERATOR EXHAUST
30.8
TREATED EFFLUENT WATER
RIVER WATER
PRECOMBUSTION AIR
11.5
LIQUID WASTE NOZZLE C
0.008
PRECOMBUSTION AIR
SOLID WASTES
AFTER-BURNER
QUENCH TOWER
VENTURI SCRUBBER
DEMISTER
WET ELECTROSTATIC PRECIPITATOR
STACK
LIQUID WASTE NOZZLE 8A
—
ROTARY KILN
RIVER WATER
LIQUID WASTE NOZZLE 8B
3.46
LOW-BTU LIQUID WASTE
—
INCINERATOR ASH
3.52
4.68
ASH PIT WATER AND SOLIDS *
0.1
WATER AND SOLIDS
52.5
WATER AND SOLIDS
12.4
WATER AND SOLIDS *
3.9

NOTE - Loadings stated in grams per year, and calculated as averages of three sampling days (8/28, 8/30, and 9/5/84).

* Sample analysis not completed for one of three sampling days. Average of two sampling days stated.

TOTAL LOADINGS OF OCDF		
In		Out
45.6	(gm/yr)	72

APPENDIX E

DETAILED DESCRIPTIONS OF AMBIENT AIR MONITORING EQUIPMENT AND SAMPLING METHODS
MICHIGAN DIOXIN STUDIES
MIDLAND, MICHIGAN, AMBIENT AIR SAMPLING STUDY

APPENDIX E

In the following narrative, each individual type of sampling device used in the ambient air study is described in terms of its components.

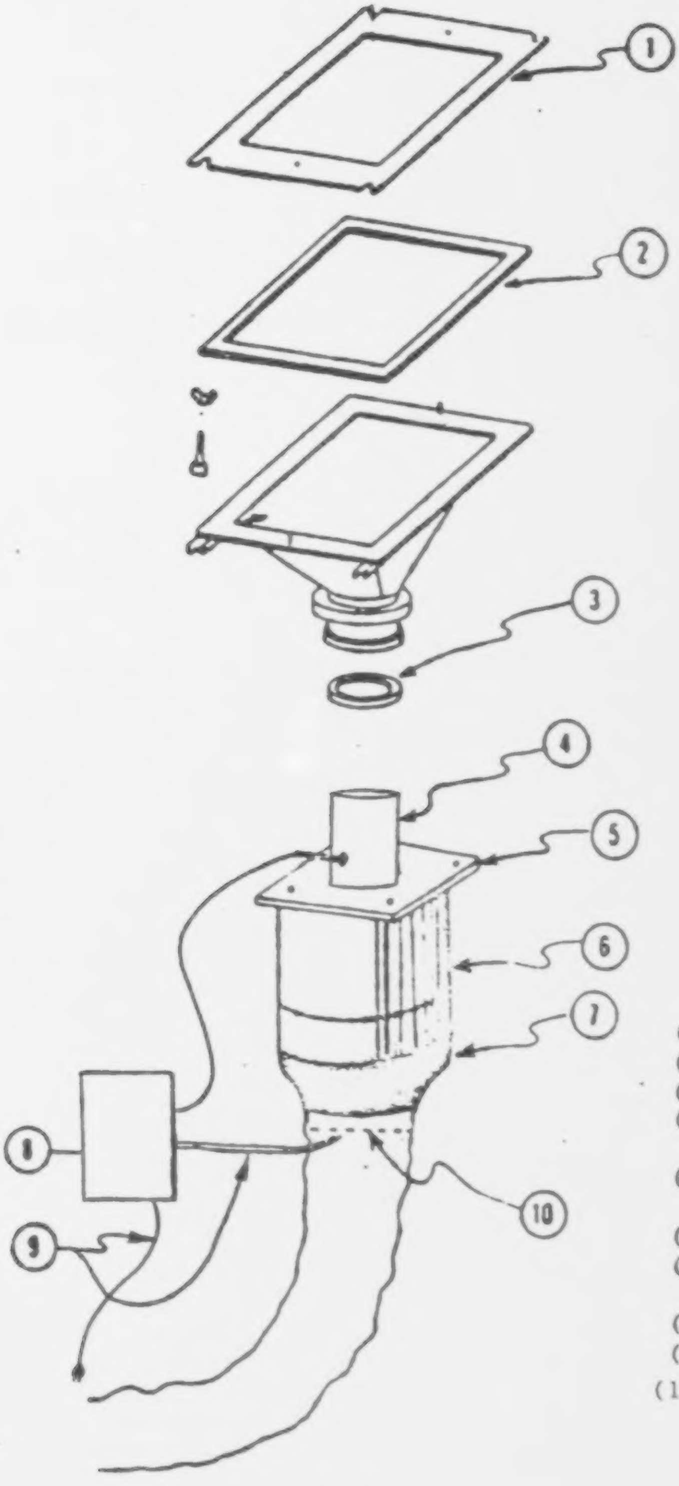
I. HIGH-VOLUME SAMPLER FOR PCDD/PCDF

Previous studies[11,12,13] showed the applicability of a modified high-volume sampler in the collection of pesticides and other semi-volatile compounds in air. More recently, the use of this sampler was extended to apply to PCDD and PCDF. The modified sampler, shown in an exploded view in Figure E-1, consisted of a high-volume sampler with a shelter, motor, timer, and flow controller arranged in a manner similar to that described in the April 30, 1971, Federal Register (Vol. 36, Number 84). However, an extended throat section was inserted between the glass fiber filter and the motor, to hold a cylindrical polyurethane foam (PUF) plug.

Standard glass fiber filters (Whatman 934-AH) of the type specified in the above Federal Register were used; that is, they were at least 99% efficient in trapping particles of 0.3-micron average diameter. Filters were used as supplied, and were not subjected to any precleaning steps. The PUF plugs were manually cut from 3-inch stock of open-cell polyether-type material, into cylindrical shapes 10 to 11 centimeters in diameter. Initial cleanup of the PUF plugs was accomplished by the field contractor, GCA/Technology Division, by Soxhlet extraction for 14 to 24 hours at four cycles per hour, three times, using 95:5 V/V hexane/ethylether. Extracted PUF was placed in a vacuum oven evacuated by a water aspirator, and dried at room temperature for two to four hours until a solvent odor was absent. Each plug was then placed in a cleaned, labeled hexane-rinsed sample container, using hexane-rinsed forceps, for transport to the sampling sites. A representative sample of every lot of cleaned PUF was analyzed at GCA for background levels of contaminants. The results of these tests are presented in Table E-1.

II. HIGH-VOLUME SAMPLER FOR CHLOROBENZENES AND OTHER SEMI-VOLATILE COMPOUNDS

Lewis and MacLeod cite[11] data indicating the collection efficiency of a sampler with PUF alone as the sorbent decreases dramatically for chlorobenzenes below Cl_5. On this basis it was decided that, to sample for semi-volatile compounds, a backup sorbent would be employed in a separate set of samplers constructed similarly to the PCDD/PCDF samplers described above. The extended throat beneath the glass-fiber filter was packed with a sorbent "sandwich" consisting of two PUF plugs of the same size as in the PCDD/PCDF sampler, surrounding a layer of 75 grams of 16/50 mesh Amberlite XAD-2 (Rohm & Haas, Philadelphia, Pennsylvania) resin. To facilitate handling of this finely divided sorbent, it was contained in a Teflon cup, as shown in Figure E-2. The



(1) Filter holder support
(2) Teflon gasket
(3) Teflon gasket
(4) Aluminum throat extension and foam plug
(5) Aluminum flange & motor support
(6) Hi-Vol motor
(7) Sheet metal adaptor for exhaust duct
(8) Flow controller & sensor
(9) Power cords
(10) Exhaust duct

FIGURE E-1

EXPLODED VIEW OF AMBIENT AIR SAMPLER FOR PCDD/PCDF

TABLE E-1

RESULTS OF QUALITY CONTROL CHECKS ON UNEXPOSED POLYURETHANE FOAM PLUGS

	Concentration (μg)*						
Sample[1]	Diethyl Phthalate	Bis 2-Ethyl Hexyl Phthalate	Adipate Alkyl Ester	Phenolics	PCDDS and Biphenyls	C_6-C_8 Hydrocarbons	Higher Boiling Point Hydrocarbons
QC 365	0	ND	ND	ND	ND	100 - 500	ND
QC 366	120.05	24	Found	ND	ND	ND	ND
QC 367	231.78	ND	ND	ND	ND	ND	ND
QC 368	53.44	ND	ND	ND	ND	ND	ND
QC 369	220.23	ND	ND	ND	ND	ND	100

*Detection Limits

Phenolics: ND = <50 μg

TCDD: ND = <100 μg

Biphenyls: ND = <100 μg

Priority pollutants = <10 - 50 μg

[1] Identity of quality control samples:

QC 365 - Laboratory blank. Solvent KD concentrated to 10 mL.
QC 366 - Two PUF plugs from Lot #1 ("Old PUF")
QC 367 - Two PUF plugs from Lot #1 ("Old PUF")
QC 368 - Two PUF plugs from Lot #7 ("New PUF")
QC 369 - Two PUF plugs from Lot #7 ("New PUF")

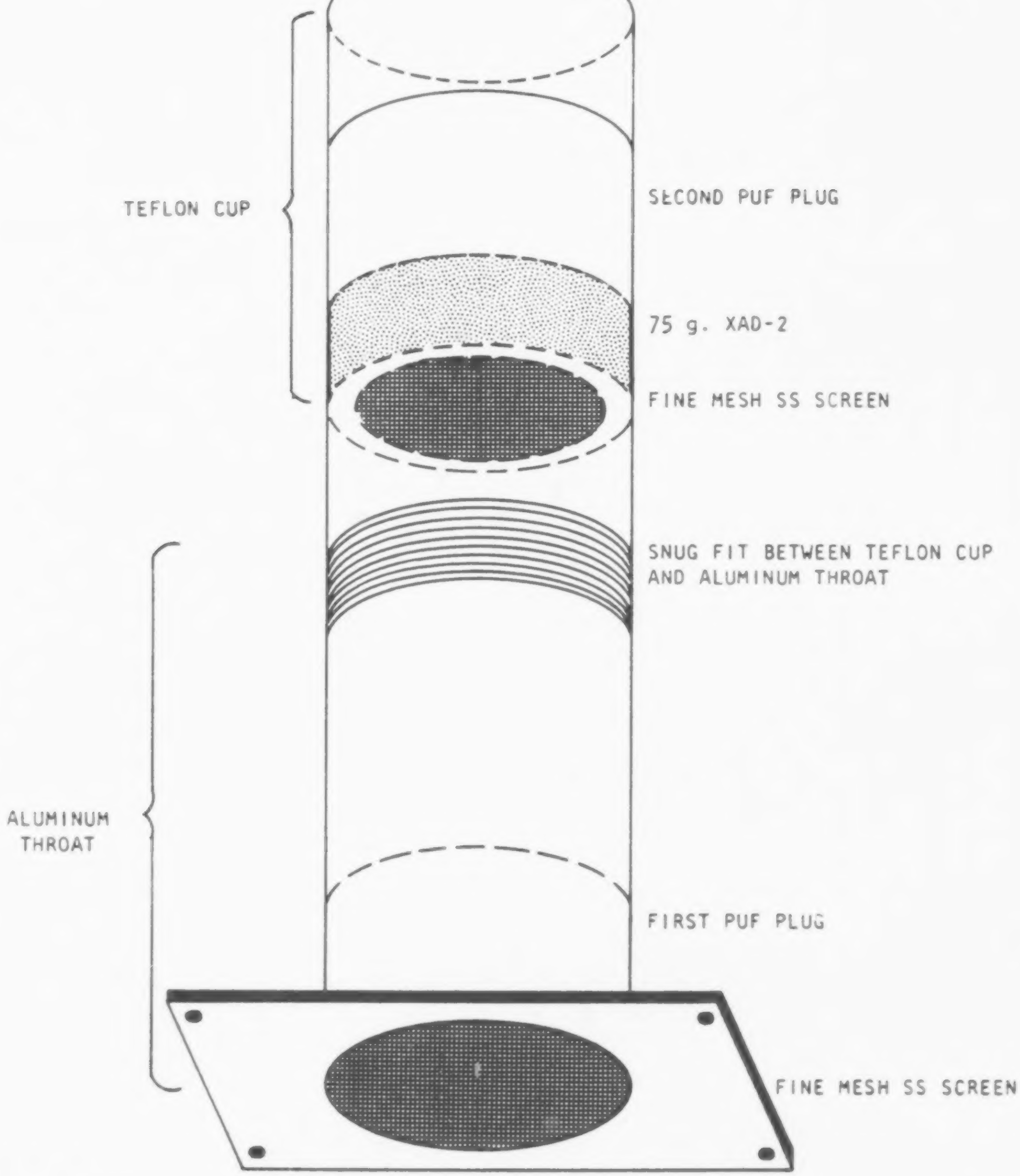

TEFLON CUP
SECOND PUF PLUG
75 g. XAD-2
FINE MESH SS SCREEN
SNUG FIT BETWEEN TEFLON CUP AND ALUMINUM THROAT
ALUMINUM THROAT
FIRST PUF PLUG
FINE MESH SS SCREEN

FIGURE E-2

HIGH-VOLUME SAMPLER ATTACHMENT
TO SAMPLE FOR CHLOROBENZENES AND OTHER SEMI-VOLATILE COMPOUNDS

cup included a 40-mesh stainless steel screen bottom, and was filled in the field with preweighed aliquots of XAD-2 delivered from containers sealed at the GCA laboratory. The sampling assembly was constructed by placing a PUF plug in the aluminum throat, a Teflon cup containing XAD-2 atop the plug, and a second PUF plug into the top of the cup. The prefilter head was secured to the top of the extended throat, forming a tightly-packed sorbent assembly.

A representative sample of every lot of XAD-2 used in this study was analyzed by the supplier, Supelco, Inc. These data are shown in Table E-2, and show the sorbent to have met requirements established by EPA for the maximum content of contaminants in unexposed sorbent.[14]

III. LOW-VOLUME SAMPLER FOR VOLATILE COMPOUNDS

As the compounds selected to be sampled in this study included several with boiling points lower than 100°C, a sampling method appropriate to the collection of these more volatile pollutants was found in the work of Riggin.[6] Carbon molecular sieve (CMS) adsorbents were determined to be appropriate to collect selected volatile organic compounds, specifically, certain nonpolar organics with boiling points between -15° and 120°C. The performance of CMS adsorbents was described by Riggin as superior to and more sensitive than other sorbents, such as Tenax GC, for a wider range of compounds. With the guidance of the document cited above, a low-volume sampler incorporating Spherocarb® adsorbent was constructed, as shown in Figure E-3.

The sampling system consisted of a pair of sorbent cartridges, each approximately three inches long, constructed of 1/4-inch O.D. stainless steel tubing. Each tube was loosely packed with 0.4 gram of 60/80 mesh Spherocarb held in place with precleaned glass wool plugs; the direction of sampled air flow was engraved on the body of the tubes to assure that the tubes were assembled correctly in sampling and analysis. The tubes were equipped with Swagelok fittings at both ends, and were prelabeled such that one tube was designated an inlet or primary tube. The primary tube was mated with a secondary or backup tube to evaluate penetration of compounds through the primary tube. The tube pair was connected by a length of Teflon tubing to a duPont model P-125 or Alpha 2 constant flow pump capable of maintaining accurately the low flow rates required (approximately 30 to 70 mL/min).

In field use, the cartridge pair was hung vertically from a support built onto one of the high-volume samplers described above. It was found that during heavy rains, water was drawn into the unprotected inlet of the primary tube. A funnel formed of aluminum foil attached to the lower end of the cartridges was successful in eliminating this problem.

To guard against the battery-powered sample pumps becoming discharged during use, they were operated while connected to battery chargers at all times. This procedure was effective in assuring reasonably constant air flows through the samplers over entire sampling periods.

TABLE E-2

QUALITY ASSURANCE ANALYSES
XAD-2 RESIN LOTS USED IN AMBIENT AIR SAMPLING

XAD-2 Lot Number	Residual Organics (ug/g)	Total Chromatographable Organics (ug/mL)
221	29.14	0.00
222	39.40	0.00
223	84.50	0.00
224	97.20	0.83
225	69.30	3.66
226	77.50	8.27
227	64.70	0.00
228	87.30	0.00
229	69.30	0.32
230	62.30	7.87

NOTE Guidelines established by EPA allow for the presence of a maximum of 1000 ug/g of residual organics, and 20 ug/mL of total chromatographable organics in unexposed sorbent media. (IERL-RTP Procedures Manual: Level 1 Environmental Assessment, 2nd Edition, EPA 600/7-78-201. U.S. Environmental Protection Agency, Research Triangle Park, NC, October 1978).

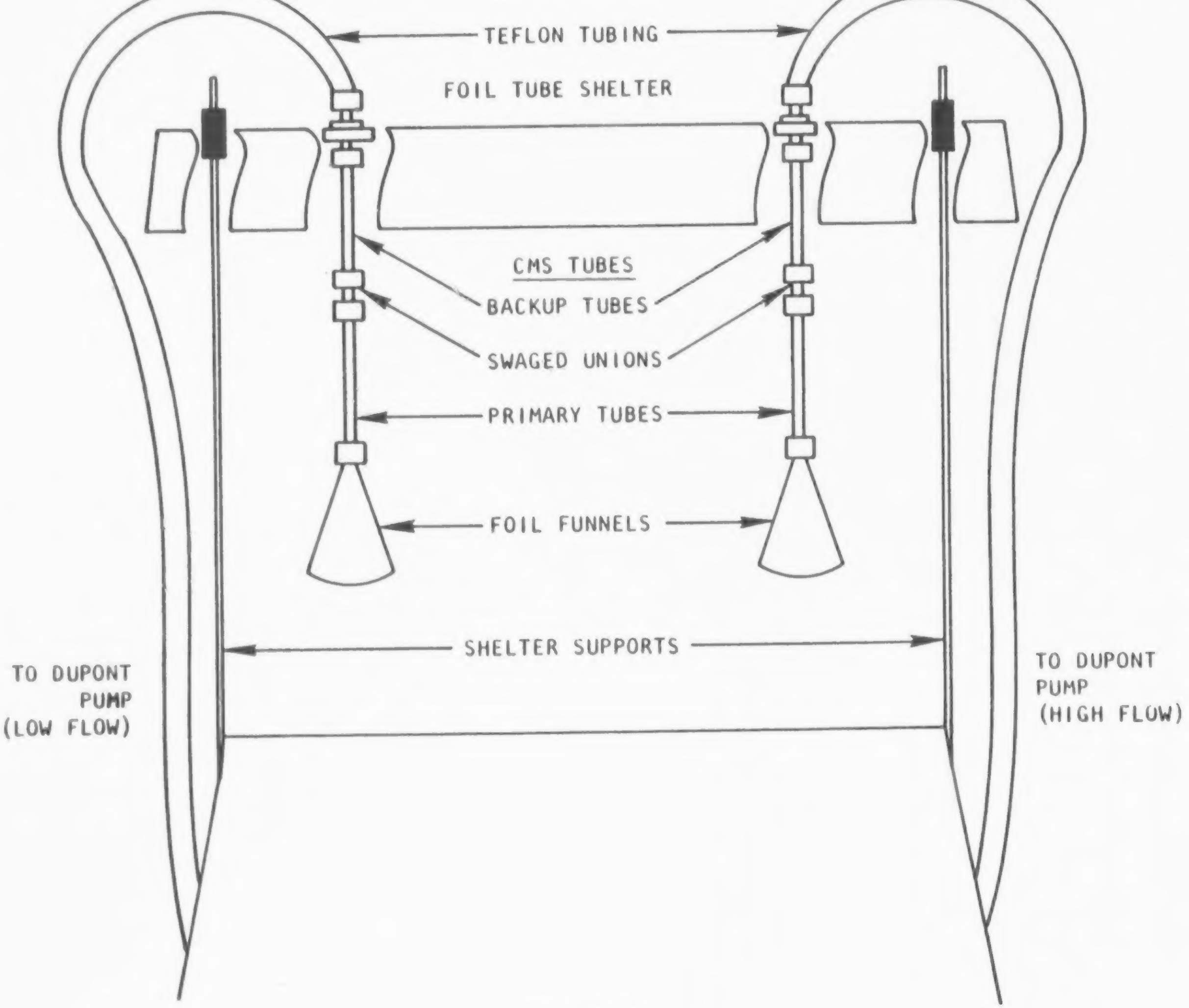

TEFLON TUBING
FOIL TUBE SHELTER
CMS TUBES
BACKUP TUBES
SWAGED UNIONS
PRIMARY TUBES
FOIL FUNNELS
SHELTER SUPPORTS
TO DUPONT PUMP (LOW FLOW)
TO DUPONT PUMP (HIGH FLOW)

FIGURE E-3

AMBIENT AIR SAMPLER FOR VOLATILE COMPOUNDS

IV. LOW-VOLUME LIQUID IMPINGER SAMPLER FOR FORMALDEHYDE

In selecting the methods to be used in sampling for the compounds of interest in ambient air, it was discovered that the solid sorbent method described above for volatile compounds was not appropriate to sample for formaldehyde, owing to apparent problems with retention on the sorbent and artifact formation. A wet chemical method involving bubbling ambient air through a mixture of 2N HCl/0.05% 2,4-dinitrophenylhydrazine (DNPH) and isooctane was chosen. Reference 6 to this report describes this method as applicable to detect aldehydes and ketones. Samples were analyzed by high-performance liquid chromatography.

The samplers (see Figure E-4) consisted of a short length of Teflon tubing connected to a pair of midget impingers, each containing the DNPH-isooctane absorbing reagent. The system was powered by a duPont constant flow sampling pump similar to that employed in the low-flow CMS sampler. The pump was joined to the impinger system by Tygon tubing. Like the CMS samplers, the inlet of the sampler was protected from rain by wrapping in a short funnel of aluminum foil.

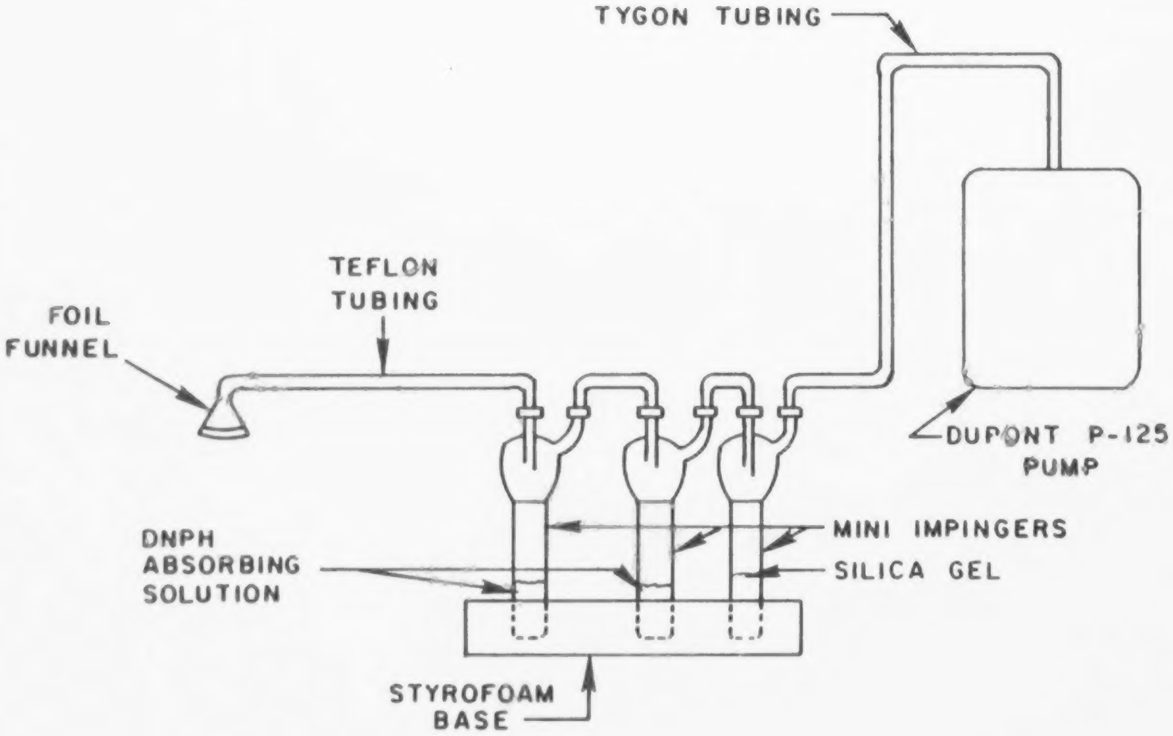

TYGON TUBING
TEFLON TUBING
FOIL FUNNEL
DUPONT P-125 PUMP
MINI IMPINGERS
DNPH ABSORBING SOLUTION
SILICA GEL
STYROFOAM BASE

FIGURE E-4

AMBIENT AIR SAMPLER FOR FORMALDEHYDE

APPENDIX F

DETAILED DESCRIPTION OF CONDUCT OF STUDY AND CARBON MOLECULAR SIEVE METHOD VALIDATION STUDY
MICHIGAN DIOXIN STUDIES
MIDLAND, MICHIGAN, AMBIENT AIR SAMPLING STUDY

APPENDIX F

I. HIGH-VOLUME SAMPLER FOR PCDD/PCDF

As indicated previously in the description of this sampler, the polyurethane foam (PUF) plugs were preextracted in the GCA laboratory, dried, and placed in a cleaned, labeled sample jar for transport to the study area. At the beginning of each sampling day, the filter supports, Teflon gaskets, and extended throats were cleaned, rinsed with hexane, and dried in a resistance-heated oven at approximately 150°C. These parts were assembled and wrapped at both ends with hexane-rinsed aluminum foil for transport to the monitoring sites. The serial numbers of the glass fiber filters were associated with the appropriate monitoring sites and recorded in a field log book maintained by the GCA field team coordinator. As the PUF plug was removed from its container and placed into the sampling assembly with hexane-rinsed forceps, the identity of the site, run number, and date of sampling was written on the exterior of the plug container.

Completed sampling assemblies were transported to the monitoring sites, where the protective foil covers were removed, and screwed tightly onto the appropriate high-volume samplers. The sampler timers were then activated and the flow controllers set to provide a target flow rate of 20 cubic feet per minute (0.57 m^3/min). In practice, however, the resistance to air flow presented by the PUF plug occasionally overcame the capacity of the sampler motor to provide this flow rate. In this case, the flow controller was set for the highest flow rate attainable. Prior to leaving each site, time, and ambient temperature, pressure, and relative humidity were recorded by the field team coordinator.

At the conclusion of each sampling run, about 24 hours later, final flow rate data were taken, the samplers were disassembled and the filter portions of the assembly were covered with hexane-rinsed aluminum foil. The samplers were then reassembled and restarted for the next sampling period. As the four monitoring sites were serviced in sequence, the sampling periods at each site were necessarily slightly different.

After each sampler was serviced, the exposed samples were returned to the mobile laboratory, where the glass-fiber filter was removed, folded inward lengthwise, and placed in a wrapper of hexane-rinsed aluminum foil. This foil was folded twice to form an envelope, labeled by filter, site, and run number, and stored flat in the mobile laboratory.

The PUF plugs were removed from the sampler assembly and returned to their original labeled container using hexane-rinsed forceps. Filter supports and the interior of the extended throats were rinsed with hexane into the PUF plug containers, and the containers were sealed for shipping to the analytical laboratory.

Sites 1, 2, and 3 were equipped with single samplers for PCDD/PCDF. On every sampling day, field blank and field duplicate samples were obtained at site 4, this being the site expected to be downwind of Dow Chemical most frequently. Method blanks, one each for the filter and the PUF, were submitted separately to the analytical laboratory; neither of these blanks was exposed to ambient air in Midland at any time with the exception of the brief period between removal of a random filter from the stock of unexposed filter media and its immediate wrapping in aluminum foil for shipment.

It was initially intended in this study to obtain PCDD/PCDF samples daily and to submit most for analysis. However, analytical cost and laboratory scheduling limitations were such that samples from three of the 18 total sampling days were analyzed. The selection was based upon examination of ambient wind data for direction and probable persistence on each sampling day. By these measures, samples from runs 4, 6, and 16 were forwarded for analysis.

II. HIGH-VOLUME SAMPLER FOR CHLOROBENZENES AND OTHER SEMI-VOLATILES

These samplers were assembled in a manner similar to that of the PCDD/PCDF units, with exceptions owing to the insertion of XAD-2 sorbent and an additional PUF plug in the high-volume sampler's extended throat (see Figure IV-7). To accomplish this, the first PUF plug was placed in the throat; its container was labeled as with the PCDD/PCDF samplers. A prewashed Teflon cup was inserted atop the first plug in the throat and filled with 75 grams of XAD-2 resin from a preweighed container; that container was also labeled appropriately according to site, run number, and date. The second PUF plug was then fitted into the top of the Teflon cup with hexane-washed forceps, and the assembly pressed together. As with the PCDD/PCDF samplers, both ends of the filter assembly were wrapped in hexane-rinsed aluminum foil for transport to the monitoring sites.

At the sites, sampler assembly was completed similarly to the PCDD/PCDF samplers. A target sampling flow rate of 20 cfm was again selected; however, this rate was achieved or exceeded during only two of the 86 successful sampler runs, because of the severe resistance to air penetration presented by the tightly-packed sorbent materials. Moreover, on some days, perhaps due to humidity, much less than the target sample volume of 800 cubic meters was collected. While runs of this kind would not have been of concern with respect to sorbent breakthrough, the sensitivity of the analytical method could have been reduced.

Following each run at each site, the sampler assembly, covered with hexane-rinsed aluminum foil at its inlet end, was dismantled, with the exposed sorbents returned to their original containers. The granular XAD-2 sorbent was poured quiescently from the Teflon cup into its container. The filter supports and throat assemblies were rinsed into the container holding both PUF plugs. Each container was then sealed for shipping.

As for PCDD/PCDF, sites 1, 2, and 3 were equipped with single samplers. Field blank and field duplicate samples were taken daily at site 4. Method blanks, one each for the filter and PUF, and of two of the ten lots of XAD-2 used in the study, were submitted for analysis for the components of interest.

Samples from each site, along with field blanks and field duplicates from site 4, were shipped for analysis for all 18 sampling days regardless of wind or other meteorological conditions.

III. LOW-VOLUME SAMPLER FOR SEMI-VOLATILES AND VOLATILES

A. CMS Field Methods

The CMS sorbent cartridges described previously were preconditioned and packed at the GCA laboratory according to the following procedure:

- Swagelok plugs, ferrules, unions, and empty stainless steel tubes were washed, rinsed with methylene chloride, and heated at 250° ± 20°C for one hour. The hardware was then assembled (see Figure IV-8).
- Each tube was packed with approximately 0.4 gram of 60/80 mesh Spherocarb and glass wool end plugs.
- Tubes were conditioned in bulk at 400°C for 16 hours under a purified nitrogen purge flow of 100 cc/min. The exit end of each cartridge was capped and the entire cartridge was removed from the flow line and the other end cap immediately installed. Sealed cartridges were then placed in a metal friction-top can containing two inches of granulated activated charcoal beneath a retaining screen. Paper tissues were placed in the can to avoid damage to the cartridges during shipment.

Tubes were conditioned in this manner no more than 30 days prior to their use in sampling.

Prior to each sampling day, two pairs of CMS tubes per sampling site were joined together by Swagelok unions. As indicated previously, the direction of air flow through the tubes was clearly labeled; thus, primary and backup tubes were designated in each pair. Sampling site identifications and run numbers were written on metal tags fastened on each individual tube. Assembled tube pairs were carried to the sampling sites in metal cans.

Each site included two low-volume samplers operating at flow rates of 30 mL/min (low-flow) and 70 mL/min (high-flow). These flow rates were selected out of concern that sorbent breakthrough volumes may have been exceeded at high sampling rates on days in which high ambient temperature and/or humidity were experienced. Prior to each sampling run, pumps were calibrated to yield sampling flow rates corresponding to the above.

At each monitoring site, a low- and high-flow pump and tube pair were assembled as shown in Figure IV-8. Pumps were started, times and meteorological data were taken, and the samplers allowed to run for about 24 hours.

At the conclusion of each run, a final flow rate check of each pump was performed; those varying by more than ±5% from initial flow rates were flagged and the sampling runs were considered invalid. Exposed CMS tube pairs were removed, their ends closed with Swagelok caps, and placed in a can for transport back to the mobile laboratory. At the laboratory, the primary and backup tubes were separated and open ends were closed tightly with Swagelok caps. Individual tubes were then placed in a can containing a two-inch bed of activated charcoal and stored in a cooler packed with ice.

Sites 1, 2, and 3 were equipped with a low- and high-flow CMS sampler on selected sampling days. Site 4 included these in addition to field duplicate samplers operating in both flow rate ranges. A single field blank, made up from an individual unexposed CMS tube, was supplied from site 4. Thus, on each sampling day 21 tubes (primary, backup, and blank) were exposed.

Analytical laboratory resources to analyze these samples were limited such that only 180 tubes could be analyzed. Thirty of these analyses were to be associated with the method validation study to be described in the Section III.B of this appendix. A reasonable analytical scheme incorporating 150 total analyses was devised, based upon ambient temperature, humidity, and wind direction on the sampling days.

Sampling days were first selected on the basis of weather forecasts available locally. If persistent winds were expected in directions likely to establish good upwind-downwind relationships between two or more sampling sites, then the CMS samplers were activated. At the conclusion of the run, if winds were favorable, 15 of the 21 tubes utilized that day were selected for analysis based on temperature and humidity conditions. If the high temperature in the sampling period exceeded 80°F, with associated high humidity, the following CMS tubes were submitted for analysis:

- All primary low-flow samples
- All backup low-flow samples
- Field blank
- Primary and backup low-flow field duplicates (site 4)
- Primary and backup high-flow samples from the two sites most closely downwind of Dow Chemical

On cooler days with lower humidity, the following tubes were to be analyzed:

- All primary high-flow samples
- All backup high-flow samples
- Field blank
- Primary and backup high-flow field duplicates (site 4)
- Primary and backup low-flow samples from the two sites most closely downwind of Dow Chemical

Samples were shipped from runs 3, 4, 6, 10, 11, 12, 15, 16, and 17, resulting in a total of 135 samples submitted for analysis.

B. CMS Method Validation Study

Because the range of compounds projected to be determined using the low-volume sampler was wide, and sufficient information concerning spiking and recovery efficiencies and breakthrough volumes on Spherocarb was not available from any previous source, a short-term laboratory validation study was conducted to test the procedure. Eight volatile compounds, as shown in Table F-1, were selected to span a range of boiling points from 37° to 173°C. The validation study consisted of two segments: determination of spiking and recovery efficiency, and validation of sampling procedures and breakthrough volumes. Spiking and ambient conditioning of prepared CMS tubes was performed by GCA, while sample analysis was conducted by a contract laboratory.

To conduct the determination of spiking efficiency, each of the compounds of interest was combined in the liquid phase in a spiking carrier matrix. A known volume was drawn with a micro liter syringe and injected into the inlet of a sorbent tube by way of a heated gas chromatography injector assembly. A total of 20 carbon molecular sieve sorbent tubes were spiked at an approximate level of 100 ng per compound of interest (concentration range - 54-82 mg/L) and analyzed by the laboratory. Five CMS tubes were spiked at an approximate level of 20 ng of each compound of interest (concentration range - 5.4-8.2 mg/L) per tube.

For validation of sampling procedures and breakthrough volumes, a system was configured to provide scrubbed (organic free), humidified air at 86°F (30°C) and 85 percent relative humidity to spiked CMS tubes attached to duPont constant flow pumps. A schematic of this system is shown in Figure F-1. These validation conditions were selected to represent the worst-case ambient temperature and humidity conditions expected to be encountered in the field during the sampling program.

A total of 30 CMS tubes were used in the validation study, allowing for a range of spiking quantities, sampling rates, and total sample volumes. These data are presented in Table F-2, and show that the tubes were divided into seven distinct sets, five of which contained five tubes each, and two of which included three and two tubes, respectively. Set 1 was spiked but not subjected to the simulated ambient sampling conditions described above; this set was intended to provide a measure of spiking and recovery efficiency alone, without considering breakthrough volumes. Sets 2 through 5 were spiked prior to being conditioned, at the air flow rates shown, for various sample volumes. Set 6, including three tubes, was conditioned but not spiked, while the two tubes in Set 7 were neither spiked nor conditioned, and were thus considered to be method blank samples.

As described in Section VI.E.3 of the report of which this appendix is a part, only four of the 30 CMS tubes in the validation study were analyzed by the contract laboratory within desired holding times. Analytical results for those four tubes showed that seven of the eight compounds shown in Table F-1 were not detected. The last compound, perchloroethylene (tetrachloroethylene), was detected, but not in consistent agreement with the known levels spiked (see Table VI-10 of report).

TABLE F-1

COMPOUNDS USED FOR VALIDATION STUDY

Compound	Boiling Point (°C)
1,1-Dichloroethylene (Vinylidene Chloride)	37
Chloroform	61.7
Carbon Tetrachloride	76.5
Acrylonitrile	77.5
Benzene	80.1
Tetrachloroethylene	121
Chlorobenzene	132
o-Dichlorobenzene	173

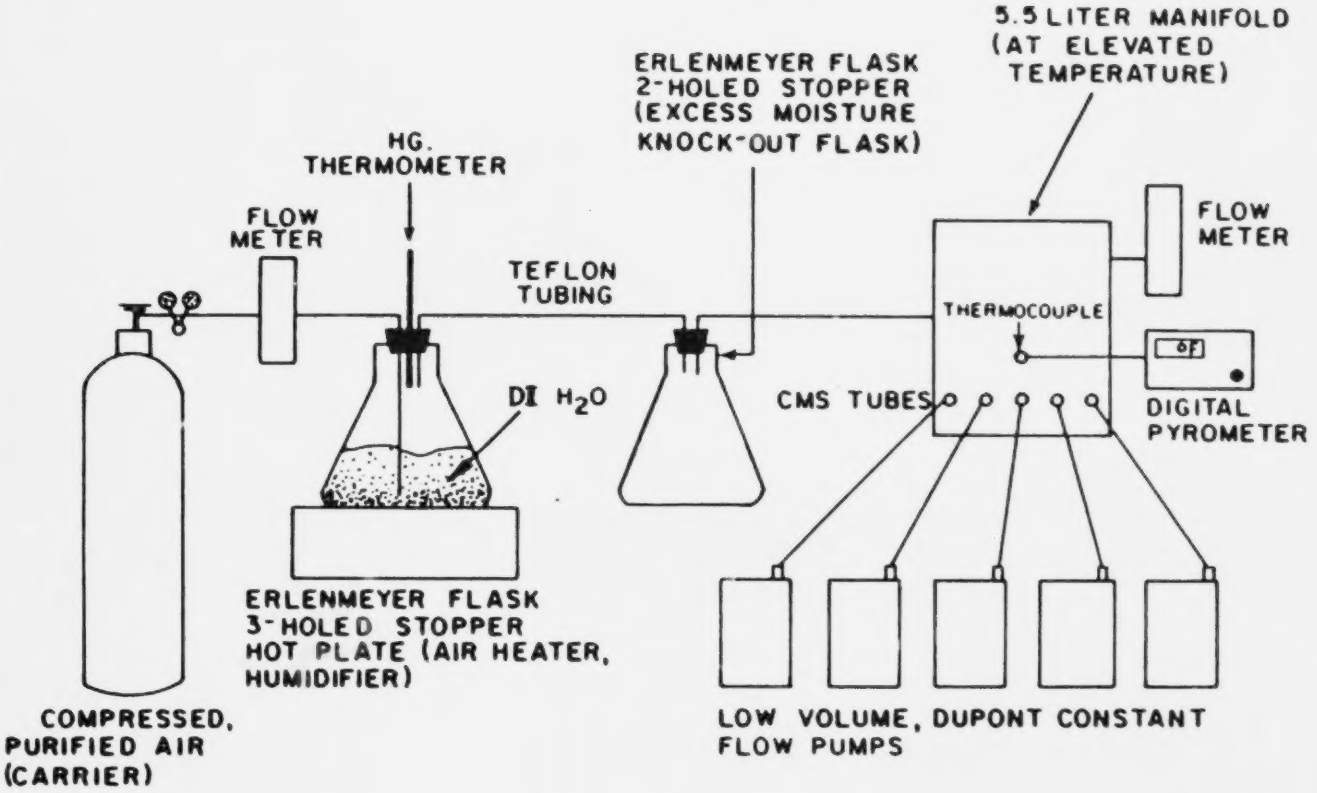

5.5 LITER MANIFOLD (AT ELEVATED TEMPERATURE)
ERLENMEYER FLASK 2-HOLED STOPPER (EXCESS MOISTURE KNOCK-OUT FLASK)
HG. THERMOMETER
FLOW METER
TEFLON TUBING
FLOW METER
THERMOCOUPLE
°F
DI H2O
CMS TUBES
DIGITAL PYROMETER
ERLENMEYER FLASK 3-HOLED STOPPER HOT PLATE (AIR HEATER, HUMIDIFIER)
COMPRESSED, PURIFIED AIR (CARRIER)
LOW VOLUME, DUPONT CONSTANT FLOW PUMPS

FIGURE F-1

SIMULATED AMBIENT AIR GENERATION SYSTEM

CMS TUBE VALIDATION STUDY

Table F-2

CMS Tube Validation Study

Tube Set Number	Run Duration (min.)	Flow Rate Average (L/min., std.)	Sample Volume (Liters, std.)	Comments
1	NA	NA	NA	No carrier air
	NA	NA	NA	Spiking level - 100 ng
	NA	NA	NA	
	NA	NA	NA	
	NA	NA	NA	
2	1,440	0.0283	40.701	Spiking level - 100 ng
	1,440	0.0271	38.980	
	1,440	0.0272	39.163	
	1,440	0.0274	39.391	
	1,440	0.0280	40.365	
3	1,440	0.0626	90.070	Spiking level - 100 ng
	1,440	0.0649	93.451	
	1,440	0.0657	94.644	
	1,440	0.0667	96.066	
	1,440	0.0642	92.462	
4	420	0.0658	27.654	7 hour run
	420	0.0623	26.162	Spiking level - 100 ng
	420	0.0629	26.410	
	420	0.0635	26.647	
	420	0.0643	26.999	
5	1,440	0.0280	40.314	Spiking level - 20 ng
	1,440	0.0276	39.723	
	1,440	0.0276	39.767	
	1,440	0.0270	38.820	
	1,440	0.0281	40.491	
6	1,440	0.0655	94.369	Blank with carrier air
	1,440	0.0646	92.980	
	1,440	0.0650	93.609	
7	NA	NA	NA	Blanks without carrier air
	NA	NA	NA	

IV. LOW-VOLUME LIQUID IMPINGER FOR FORMALDEHYDE

Sampling trains composed of the parts described in Section IV of Appendix E were assembled as shown in Figure E-4. Samples were collected and handled according to the protocols outlined in pages 40 to 43 of Reference 19 to this report. Owing to limitations on the number of samples that could be analyzed by the contract laboratory, and the requirement that DNPH absorbing reagent be used for sampling within 48 hours of its initial preparation, it was determined that samples for formaldehyde would be obtained on six of the 18 sampling days which encompassed the ambient air study period. The DNPH reagent was prepared in the GCA laboratory and air-shipped to the Midland sampling sites by commercial carrier, when requested by field contractor representatives based upon predictions of favorable wind directions.

APPENDIX G

RAW ANALYTICAL DATA
AMBIENT AIR PCDD/PCDF SAMPLING
IN VICINITY OF DOW CHEMICAL COMPANY, MIDLAND, MICHIGAN

ANALYTICAL LABORATORY - MIDWEST RESEARCH INSTITUTE
KANSAS CITY, MISSOURI

TABLE G-1

Raw PCDD/PCDF Analytical Data
Ambient Air Study in Vicinity of
Dow Chemical Company, Midland, Michigan

MRI Sample No.	SAS Sample No.	Total TCDF	2,3,7,8-TCDF	Total TCDD	2,3,7,8-TCDD	Total P_5CDF	Total P_5CDD	Total HxCDF	Total HxCDD	Total HpCDF	Total HpCDD	Total OCDF	Total OCDD
1149E-1-NFA-1	1149E-1	1.0	1.0[b]	1.3	0.75[b]	ND (0.20)[c]	0.42[b]	ND (0.19)	8.5[e]	ND (0.74)	5.0[e]	5.6[f]	5.8[f]
1149E-2-NPA-2	1149E-2	1.0	1.0[b]	1.5	0.93[b]	ND (0.46)	0.56[b]	ND (0.18)	7.3[e]	ND (0.73)	4.7[e]	5.2[f]	6.2[f]
1149E-3-FA-3	1149E-3	ND (0.11)	ND (0.11)	0.88	ND (0.10)	ND (0.09)	ND (0.16)	ND (0.13)	0.72	ND (0.52)	0.61	0.75	0.87
1149E-4-PA-4	1149E-4	0.65	ND (0.06)	0.80	ND (0.10)	ND (2.0)	ND (0.29)	ND (0.30)	ND (0.64)	ND (0.66)	ND (0.41)	ND (0.51)	0.53
1149E-5-FA-5	1149E-5	36	ND (0.69)	3.7	ND (0.10)	4.0	ND (0.48)	2.3	ND (0.69)	4.1	1.7	2.8	5.1
1149E-6-PA-6	1149E-6	180	ND (0.40)	33	ND (0.70)	28	6.0	ND (1.1)	ND (0.51)	ND (0.74)	ND (0.36)	ND (0.49)	1.2
1149E-7-FA-7	1149E-7	7.5	ND (0.20)	1.6	ND (0.18)	0.25	ND (0.38)	ND (0.30)	ND (0.26)	ND (0.65)	1.7	0.47	6.0
1149E-8-PA-8	1149E-8	3.9	ND (0.28)	1.7	ND (0.12)	3.2	ND (0.25)	ND (0.25)	ND (0.20)	ND (0.45)	ND (0.35)	0.65	1.2
1149E-9-FA-9	1149E-9	1.2	ND (0.09)	0.76	ND (0.07)	0.91	ND (0.07)	ND (0.51)	0.67	ND (0.28)	0.78	1.3	2.1
1149E-10-PA-10	1149E-10	d	d	0.84	ND (0.02)	ND (0.12)	ND (0.03)	ND (0.18)	ND (0.15)	ND (0.41)	ND (0.46)	ND (0.43)	ND (1.5)
1149E-11-FA-11	1149E-11	ND (0.03)	ND (0.03)	0.65	ND (0.05)	ND (0.07)	ND (0.11)	ND (0.48)	ND (0.35)	ND (0.85)	ND (0.88)	ND (0.98)	ND (1.6)
1149E-12-PA-12	1149E-12	ND (0.03)	ND (0.03)	0.28	ND (0.08)	ND (0.10)	ND (0.13)	ND (0.16)	ND (0.11)	ND (0.70)	ND (0.32)	ND (0.21)	0.70
1149E-13-FA-13	1149E-13	0.92	ND (0.06)	0.70	ND (0.08)	ND (0.67)	ND (0.24)	0.57	ND (0.24)	ND (0.43)	1.2	ND (0.66)	3.2
1149E-14-PA-14	1149E-14	4.0	ND (0.13)	0.61	ND (0.12)	1.1	ND (0.20)	ND (0.11)	ND (0.87)	ND (0.90)	ND (0.16)	ND (0.98)	ND (0.26)
1149E-MB1-15	-	ND (0.09)	ND (0.09)	0.84	ND (0.02)	ND (0.12)	ND (0.03)	ND (0.04)	ND (0.06)	ND (0.21)	ND (0.26)	ND (0.17)	ND (0.40)
1149E-39-MNF-16	1149E-39	1.0	1.0[b]	1.5	0.85[b]	ND (0.09)	0.36[b]	ND (0.11)	6.2[e]	ND (0.38)	4.9[e]	7.8[f]	7.0[f]
1149E-15-FB-17	1149E-15	5.6	ND (0.10)	1.1	ND (0.06)	ND (0.06)	ND (0.11)	ND (0.49)	ND (0.80)	ND (0.09)	2.3	0.78	1.3
1149E-16-FB-18	1149E-19	5.4	ND (0.11)	0.94	ND (0.06)	ND (0.36)	ND (0.61)	ND (0.19)	ND (0.13)	ND (0.16)	0.50	0.62	3.9
1149E-18-FB-19	1149E-21	2.2	ND (0.11)	1.3	ND (0.13)	0.58	ND (0.24)	ND (0.14)	ND (0.12)	ND (0.13)	0.18	ND (0.42)	2.1
1149E-19-FB-20	1149E-23	ND (0.05)	ND (0.03)	1.0	ND (0.08)	ND (0.06)	ND (0.05)	ND (0.01)	ND (0.01)	ND (0.07)	ND (0.05)	ND (0.10)	ND (0.20)
1149E-20-FB-21	1149E-25	1.5	ND (0.05)	0.66	ND (0.08)	1.0	ND (0.38)	ND (0.23)	ND (0.82)	ND (0.08)	0.28	ND (0.25)	3.3
1149E-40-NMP-22	1149E-40	0.82	0.82[b]	1.3	0.83[b]	ND (0.03)	1.0[b]	ND (0.09)	3.4[e]	ND (0.12)	3.1[e]	4.1[f]	7.1[f]
1149E-15-PB-23	1149E-16	5.8	ND (0.14)	0.77	ND (0.15)	ND (2.3)	ND (0.30)	ND (0.20)	ND (0.45)	ND (1.7)	ND (0.22)	ND (0.72)	ND (2.4)
1149E-16-PB-24	1149E-18	6.9	ND (0.15)	1.8	ND (0.15)	ND (0.67)	ND (0.27)	ND (0.64)	ND (0.48)	ND (1.2)	ND (2.2)	ND (1.4)	ND (4.2)
1149E-17-PB-25	1149E-20	29	ND (0.18)	6.0	ND (0.82)	1.7	ND (0.39)	ND (1.0)	ND (0.91)	ND (0.95)	ND (0.91)	ND (1.2)	ND (4.0)
1149E-18-PB-26	1149E-22	8.7	ND (0.16)	0.74	ND (0.12)	0.25	ND (0.12)	ND (1.0)	2.3	ND (0.71)	1.7	3.6	5.4
1149E-19-PB-27	1149E-24	ND (0.41)	ND (0.10)	3.5	ND (0.61)	ND (0.79)	ND (0.10)	ND (0.31)	ND (0.89)	ND (0.77)	0.71	1.5	2.2
1149E-20-PB-28	1149E-26	5.1	ND (0.10)	0.57	ND (0.10)	0.78	ND (0.25)	ND (0.47)	ND (0.08)	ND (3.2)	ND (0.96)	ND (2.0)	ND (2.3)
1149E-17-FB-29	1149E-17	2.2	ND (0.04)	0.43	ND (0.03)	ND (0.03)	ND (0.09)	ND (0.60)	ND (1.6)	ND (0.96)	ND (1.2)	ND (2.1)	ND (2.1)
1149E-MB2-30	-	ND (0.04)	ND (0.04)	0.26	ND (0.03)	ND (0.18)	ND (0.21)	ND (0.35)	ND (0.28)	ND (0.51)	ND (0.56)	ND (0.59)	ND (0.67)
1149E-41-NMF-31	1149E-41	0.94	0.94[b]	1.3	0.87[b]	ND (0.04)	0.55[b]	ND (0.14)	5.1[e]	ND (0.35)	4.6[e]	7.3[f]	8.2[f]
1149E-21-FC-32	1149E-27	3.0	ND (0.06)	0.29	ND (0.03)	ND (0.09)	ND (0.22)	ND (0.24)	ND (0.17)	ND (0.53)	ND (0.64)	ND (1.0)	ND (2.3)
1149E-22-FC-33	1149E-29	58	ND (0.81)	4.5	ND (0.03)	2.2	ND (0.25)	3.7	0.45	2.4	2.2	1.1	6.2
1149E-23-FC-34	1149E-31	4.6	ND (0.10)	0.71	ND (0.07)	ND (0.09)	ND (0.29)	ND (0.13)	ND (0.33)	ND (0.46)	0.46	ND (0.53)	2.3
1149E-24-FC-35	1149E-33	72	ND (1.1)	9.3	ND (0.20)	6.3	ND (0.45)	2.4	0.22	2.4	0.91	3.7	3.2

TABLE G-1 (continued)

MRI Sample No.	SAS Sample No.	Total TCDF	2,3,7,8-TCDF	Total TCDD	2,3,7,8-TCDD	Total P_5CDF	Total P_5CDD	Total HxCDF	Total HxCDD	Total HpCDF	Total HpCDD	Total OCDF	Total OCDD
1149E-25-FC-36	1149E-35	4.5	ND (0.10)	1.1	ND (0.12)	ND (0.10)	ND (0.36)	ND (0.10)	ND (0.25)	ND (0.69)	ND (0.33)	ND (0.58)	ND (0.72)
1149E-26-FC-37	1149E-37	100	ND (1.1)	20	ND (0.46)	12	ND (0.91)	3.4	0.75	2.1	1.1	5.1	3.4
1149E-21-PC-39	1149E-28	3.8	ND (0.10)	0.78	ND (0.06)	ND (0.12)	ND (0.12)	ND (0.16)	ND (0.12)	ND (0.32)	ND (0.37)	0.12	0.28
1149E-22-PC-40	1149E-30	82	ND (0.10)	19	ND (0.04)	3.9	ND (0.26)	ND (0.40)	ND (0.09)	ND (0.56)	ND (0.32)	0.21	5.5
1149E-23-PC-41	1149E-32	9.7	ND (0.04)	1.6	ND (0.06)	ND (0.19)	ND (0.40)	ND (0.06)	ND (0.12)	ND (0.67)	ND (0.09)	0.59	ND (0.77)
1149E-42-NMP-38	1149E-42	3.6	0.92[b]	1.7	1.2[b]	ND (0.06)	0.47	ND (0.06)	4.1[e]	ND (0.29)	5.4[e]	7.5[f]	8.0[f]
1149E-24-PC-42	1149E-34	240	ND (1.3)	52	ND (1.3)	23	1.09	ND (1.8)	ND (3.4)	ND (0.74)	ND (1.3)	ND (0.96)	ND (1.2)
1149E-25-PC-43	1149E-36	8.0	ND (0.15)	1.1	ND (0.07)	ND (0.07)	ND (0.12)	ND (0.46)	ND (0.70)	ND (2.4)	ND (1.5)	ND (0.80)	ND (1.7)
1149E-26-PC-44	1149E-38	5.1	ND (0.25)	g	g	ND (0.30)	ND (0.30)	ND (0.19)	ND (0.31)	ND (1.0)	ND (1.0)	ND (0.98)	ND (2.5)
1149E-MB3-45	-	ND (0.02)	ND (0.02)	0.44	ND (0.04)	ND (0.03)	ND (0.05)	ND (1.0)	ND (2.0)	ND (0.61)	ND (1.4)	ND (1.3)	ND (3.0)

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a All data reported as nanograms (ng)/sample.
b Sample originally spiked with 1 ng of this compound.
c Value in parenthesis reflects estimated detection limit.
d Sample analyzed after additional cleanup by carbon column. TCDD and TCDF internal standards not recovered.
e Sample originally spiked with 5 ng of a single isomer.
f Sample originally spiked with 10 ng of a single isomer.
g The 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard was not recovered. Calculations for TCDF, P_5CDF and P_5CDD based on 2,3,7,8-TCDF-$^{13}C_{12}$.

TABLE G-2

Key to Sample Identification
Ambient Air PCDD/PCDF Sampling
In Vicinity of Dow Chemical Company, Midland, Michigan

SAS Sample Number (1149E-	Sample Identity
1	9/8-9/84; Glass Fiber Filter (GFF) Method Blank
2	9/8-9/84; Polyurethane Foam (PUF) Method Blank
3	9/8-9/84; Site 1 GFF
4	9/8-9/84; Site 1 PUF
5	9/8-9/84; Site 2 GFF
6	9/8-9/84; Site 2 PUF
7	9/8-9/84; Site 3 GFF
8	9/8-9/84; Site 3 PUF
9	9/8-9/84; Site 4 GFF
10	9/8-9/84; Site 4 PUF
11	9/8-9/84; Site 4 Field Blank GFF
12	9/8-9/84; Site 4 Field Blank PUF
13	9/8-9/84; Site 4 Field Duplicate GFF
14	9/8-9/84; Site 4 Field Duplicate PUF
15	9/12-13/84; Site 1 GFF
16	9/12-13/84; Site 1 PUF
17	9/12-13/84; Site 2 GFF
18	9/12-13/84; Site 2 PUF
19	9/12-13/84; Site 3 GFF
20	9/12-13/84; Site 3 PUF
21	9/12-13/84; Site 4 GFF
22	9/12-13/84; Site 4 PUF
23	9/12-13/84; Site 4 Field Blank GFF
24	9/12-13/84; Site 4 Field Blank PUF
25	9/12-13/84; Site 4 Field Duplicate GFF
26	9/12-13/84; Site 4 Field Duplicate PUF
27	9/22-23/84; Site 1 GFF
28	9/22-23/84; Site 1 PUF
29	9/22-23/84; Site 2 GFF
30	9/22-23/84; Site 2 PUF
31	9/22-23/84; Site 3 GFF
32	9/22-23/84; Site 3 PUF
33	9/22-23/84; Site 4 GFF
34	9/22-23/84; Site 4 PUF
35	9/22-23/84; Site 4 Field Blank GFF
36	9/22-23/84; Site 4 Field Blank PUF
37	9/22-23/84; Site 4 Field Duplicate GFF
38	9/22-23/84; Site 4 Field Duplicate PUF
39	9/12-13/84; GFF Method Blank
40	9/12-13/84; PUF Method Blank
41	9/22-23/84; GFF Method Blank
42	9/22-23/84; PUF Method Blank

APPENDIX H

RESULTS OF REANALYSIS OF SELECTED PCDD/PCDF SAMPLES BY USEPA-EMSL-RTP AND EXPLANATORY INFORMATION

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NORTH CAROLINA 27711

DATE: MAY 2,1985

SUBJECT: ANALYSIS FOR CDDs AND CDFs IN EXTRACTS OF AMBIENT AIR

FROM: ROBERT L. HARLESS, RESEARCH CHEMIST Robert Harless
METHODS DEVELOPMENT BRANCH/EMSL-RTP (MD-77)

TO: Dr. NORBERT JAWORSKI, DIRECTOR
ENVIRONMENTAL RESEARCH CENTER-DULUTH and
HQ LIASION FOR NATIONAL DIOXIN STUDY

Background information regarding these analyses is briefly summarized. Ambient air samples were collected in Region V utilizing high volume air samplers. The samples were subjected to Midwest Research Institute (MRI) extraction and clean-up procedures and analyzed for CDDs and CDFs. Region V then requested that MRI submit specific sample extracts and analytical standards to EMSL-RTP for confirmatory analysis. High levels of TCDFs were the main concern. Details were discussed in a conference call with Curtis Ross and Frank Thomas, Region V, Dr. Norbert JAWORSKI, ERL-D and myself.

Sixteen sample extracts, a labeled CDD analytical standard and a native CDD/CDF analytical standard were received from MRI on 3/1/1985. HRGC-HRMS analyses were performed on the standards and extracts utilizing a 60m SP-2330 fused silica capillary column for resolution of components. The concentrations of MRI analytical standards were compared with EMSL-RTP and ECL analytical standards. Four extracts specified by Region V were subjected to analysis for TCDDs,TCDFs and penta-CDFs as requested. Preliminary analytical results were discussed with Frank Thomas, Region V, in mid March at which time I indicated that this report would not be written until a TCDF isomer that was needed for identification purposes was received. The work is now complete. Analytical results are shown in Table 1, and summarized below.

* The stated concentration of labeled and native 2378-TCDD and TCDF in MRI standards are in reasonable agreement with concentrations of ECL and EMSL-RTP standards.

* MRI standards were used for quantification purposes. Comparisons of MRI and EMSL-RTP results indicate that in general most values agree from the standpoint of low or high amounts in each extract.

* The high amounts of TCDF in the extracts is due to one or more of the following isomers, 1238-,1467-,2468-,1236-TCDF that elute simultaneously from a 60m SP-2330 fused silica capillary column. A 2468-TCDF isomer was obtained for identification purposes. The retention time is within acceptable agreement, one

second, with the isomer or isomers in the extracts. The TCDFs in the extracts by themselves are unusual. Dr. Rappe's work in ES and T, Vol.18,no.3, 1984 was used for reference purposes since I do not have these four individual isomers. Many isomers including these are found in effluents from incineration processes. Also, some are present in chemical products. For example, 2468-TCDF is an impurity in 246-trichlorophenol.

* Several extracts of soil from the study performed last year were analyzed again to determine if this specific TCDF or TCDF isomers were present. The analyses of 13394 indicates that the same isomer or group of isomers is also present in the soil extract.

* The distribution of TCDD isomers in the extracts of ambient air is also similar to those found in effluents from incineration processes and the extracts of soil that were analyzed in the study last year. However, there are some differences in the ratio of various isomers in the extracts of ambient air.

* Chlorinated diphenylethers are responsible for some (20 to 50%) of the concentration reported as penta-CDFs in the extracts. Region V did not request or instruct MRI to perform this analysis required to differentiate CDFs from chlorinated diphenylethers. It should be done in future studies.

In summary, the TCDFs,TCDDs,and PCDFs present in extracts of ambient air were also present in the extracts of soil from the general area that were analyzed in the study last year. The distribution of TCDD isomers is similar to those found in effluents from incineration processes. The TCDF isomer or isomers by themselves are not similar to those found in incineration processes. However they were present in the extracts of soil that were analyzed last year. The amounts of 2378-TCDD and 2378-TCDF in the ambient air extracts are very low and or not detected in most cases. Evaluation of the data indicates that the TCDFs,TCDDs, and PCDFs in the ambient air extracts may be due to: (1) airborne particulate matter from incineration processes on a daily basis or (2) contaminated soil in the area that became airborne during the time that the air sampler was in operation. The air sampler collection and retention efficiency for CDDs and CDFs has not been validated. Therefore, results should be considered as minimum values and actual maximum values are unknown.

The MRI extracts and analytical standards are stored for reference. Please call me if you have any questions.

CC:C.ROSS
M.DELLARCO
N.WILSON
J.CLEMENTS
R.LEWIS

TABLE 1. ANALYTICAL RESULTS FOR TCDFs AND TCDDs IN EXTRACTS OF AMBIENT AIR

COMPOUNDS	SAMPLE ID AND ESTIMATED AMOUNTS (ng) IN THE EXTRACTS			
	1149E-5	1149E-6	1149E-7	1149E-8
2378-TCDF"RT"[a]	0.2	-	-	0.4
TOTAL TCDFs	28.0	131.0	2.2	26.0[b]
2378-TCDD	0.4	-	-	-
TOTAL TCDDs	9.0	29.0	0.8	1.4

a The concentrations shown above for 2378-TCDF are for the specific time window exhibited by 2378-TCDF analytical standard. However, NOTE, conclusive assignment of 2378-TCDF in these extracts can not be made because the other two TCDF isomers required for conclusive identification purposes are not available.

b Average of two analyses performed on separate days.

Refer to text for comments regarding these analyses.

APPENDIX J

DETAILED DISCUSSION OF AIR DISPERSION MODELING TO DETERMINE POINT OF MAXIMUM GROUND-LEVEL IMPACT

Possible Association of Stack Emissions and Ambient Monitored Concentrations of CDDs/CDFs

A fundalmental question arises as to the possible origin of the ambient monitored concentrations of CDDs/CDFs in Midland, Michigan. Dow Chemical Company has concluded that, "dispersion of ashes and vent stack particulates from historical incineration operations are the probable source of the trace TCDD levels now found in the local (Midland) environment (Dow, 1984)." This qualitative conclusion was reached by comparing current emissions of 2378-TCDD with levels measured in the ambient air and in Midland soil. An independent panel of experts reviewed the Dow report and concluded: "The major identified source of 2378-TCDD into the air and soil of the Midland area is the waste incinerator stack with an estimated release of 0.33 gm of 2378-TCDD per year (Cooks, 1984)." However, the relative magnitude of these emissions overtime was brought into question by the expert panel,

> "The conclusions are based on just two samples of stack particulates and three samples of incinerator ash. Given the variable nature of the feed to the incinerator, this represents a weakness in the study (Cooks, 1984)."

Apparently Dow never investigated a possible ground level impact of CDD/CDF emissions through the use of accepted air dispersion models. This would have permitted a more complete analysis of the predicted ambient concentration of CDDs/CDFs resulting from stack dispersion verses the concentration of CDDs/CDFs measured with ambient monitors. The EPA's Human Exposure Model predicted the maximum annual average concentration of 2378-TCDD equivalence emitted from the waste incinerator stack to occur approximately one kilometer northeast and east-northeast downwind of the facility. This agrees qualitatively with the placement of sampling Stations 2 and 4 for ambient air sampling in Midland, Michigan. Station 2 is approximately 1.3 km northeast of the incinerator, and Station 4 is approximately 1.8 km east-northeast of the incinerator. Station 2 measured approximately 3.5 pg 2378-TCDD equivalence/m^3 of air, and Station 4 measured about 2 pg 2378-TCDD equivalence/m^3 of air. The dispersion model predicted about 0.10 pg 2378-TCDD equivalence/m^2 of air, however, this concentration reflected five years of average meteorology. In addition, the mass emission rate of 2378-TCDD equivalence was an average emission rate over three days of stack sampling. The ambient monitoring was not conducted in concert with the stack testing, therefore, it is likely there would be no perfect correlation between the relative magnitude of the predicted concentration and the ambient monitored concentration. The apparent agreement with the predicted fallout area using dispersion modeling and the location of the ambient air samplers does suggest that current emissions from the waste incinerator may be contributing to measurable concentrations of CDDs/CDFs on the ground.

Site 4 was chosen to compare the percent distribution of CDDs/CDFs measured in the ambient with the percent distribution of CDDs/CDFs emitted from the waste incinerator. Table 4 summarizes this comparison. The average distribution of CDDs and CDFs relative to the total CDDs or CDFs in monitoring Station 4, when compared to the average distribution in incinerator

TABLE 4. Average Percent Distribution of CDDs and CDFs in Both Stack Emissions of the Dow Incinerator and Ambient Monitored Concentrations in Midland, MI.

Pollutant	Incinerator[a] Emissions (Percent)	Station 4[b] Ambient Monitoring (Percent)
2378-TCDD	0.9	0.38
TotalTCDD	86.06	37.00
PentaCDD	7.72	Not Reported
HexaCDD	1.15	13.88
HeptaCDD	1.03	10.91
OctaCDD	3.10	34.41
2378-TCDF	1.70	0.78
Total TCDF	86.37	71.57
PentaCDF	9.04	14.13
HexaCDF	2.41	0.24
HeptaCDF	0.45	0.24
OctaCDF	0.13	13.07

NOTE: Percent distribution is determined by $\frac{\text{CDD}}{\text{Total CDD}}$, and $\frac{\text{CDF}}{\text{Total CDF}}$.

(a) Incinerator distribution was determined as an average of EPA stack tests on 8/28 and 8/30.

(b) Average distribution of 3 sampling days at Station 4.

emissions, does suggest that the incinerator emissions may be contributing to the CDDs and CDFs measured by the monitor. For example, 2378-TCDD is less than 1% of total CDD emissions in both incinerator emissions and in ambient measured concentrations. Total TCDD isomers predominate in both sampling regimes (incinerator emissions and ambient monitoring). OctaCDD is about 34% of CDD concentration measured in the ambient, whereas OctaCDD is only about 5% of incinerator emissions of CDDs. This may suggest atmospheric transformation in the isomer ratios of the incinerator emissions as the CDDs are dispersed from the stack to the ground. However, this is only speculation since such phenomena are currently poorly understood, and have only recently been hypothesized (Czucwa, 1986). In any case, given the fact that ambient sampling and stack testing occurred over different time periods, there is relatively good agreement in the homologue distribution patterns of the two sampling regimes. A similarity in distribution of CDFs can also be seen. For example, 2378-TCDF is a minor constituent ($\leq$ 1%) of the total CDFs measured in incinerator stack emissions and in ambient monitoring. The predominant CDF in both measurements is total TCDF isomers. PentaCDF isomers constitute the second most frequent isomers in emissions of CDFs in both incinerator emissions and in the ambient concentrations.

Although there is not a perfect comparison in the distribution pattern of CDFs/CDDs, there appears to be relatively good agreement between the two sampling regimes to suggest a continued contribution of the waste incinerator to ambient concentrations of CDDs/CDFs in the Midland, Michigan, environment. The measured ambient concentrations confirm the significance of even low levels of emissions from stationary combustors, if these levels are a daily occurrence, and continue over a long period of time.

This "fingerprint" analysis in which the area of maximum fallout from incinerator emissions, and the percent distribution of CDDs/CDFs in incinerator emissions is compared with the ambient measured concentrations, can only suggest an association between the incinerator and ambient levels of CDDs/CDFs in Midland. Perhaps a rigorous analysis of emissions using micrometeorology recorded for the nearby nuclear power plant project would help resolve a quantitative association. In addition, morphological comparisons of particulate matter emitted from the waste incinerator to particulate matter captured in ambient samplers could also help resolve a quantitate association between emissions and ambient levels in Midland. Electron microscopy could aid such an analysis.

This report cannot rule out the possibility that sources other than, or in addition to, CDD/CDF emissions from the waste incinerator may be contributing to CDDs/CDFs measured at ambient monitoring stations in Midland. These possibilities include: fugitive process emissions during chemical manufacturing at Dow; fugitive emissions at both the electrical powerhouse and waste incinerator at Dow, and re-entrainment of contaminated soil and dust particles.